# AMERICAN JOURNAL OF PHARMACY

PUBLISHED MONTHLY BY THE

Philadelphia College of Pharmacy



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Vol. 90 APRIL, 1918 No. 4


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Price, $2.00 per Annum, in advance. Issued in monthly numbers of not less than 48 pages. Single Numbers, 20 Cents. Back Numbers, 30 Cents.

Address all communications

For the Editor: To American Journal of Pharmacy, Philadelphia College of Pharmacy, Tenth Street, Philadelphia, Pa.

For the Business Manager: To Harold J. Leidy, Business Mgr., Philadelphia College of Pharmacy, Tenth Street, Philadelphia, Pa.

Publication Office—43 North Queen Street, Lancaster, Pa.

Entered as second-class matter February 14, 1879, at the Post Office at Lancaster, Pa., under Act of March 3, 1879.

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**ESTABLISHED IN 1825**

Four preliminary numbers were published at different times until in 1829, when the publication of the regular volumes began. Since then the publication has been uninterrupted. During the period from 1829 to 1852 four numbers were published annually, except in 1847, when five numbers were published. From 1853 to 1870 six numbers were published. Since this time twelve numbers have been published annually.

Manuscripts should be sent to the Editor. It should be stated in this connection that the Editor does not assume any responsibility in connection with the views or investigations of contributors, other than to exercise general care in the selection of matter.

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THE AMERICAN
JOURNAL OF PHARMACY

*APRIL, 1918*

## EDITORIAL.

### The Progress of Prerequisite Legislation.

During the past winter, two of the legislatures in session have passed educational prerequisite amendments to their pharmacy laws. As usual, these laws were enacted through the efforts of the state pharmaceutical associations. The pharmacists of Virginia and of New Jersey are to be congratulated upon this achievement in behalf of the professional elevation of pharmacy.

New Jersey lays claim to having been the first state in which the druggists formed a permanent state organization for the betterment of the conditions in their calling. As early as 1895, through the efforts of this state association a pharmacy law was enacted providing for the appointment of a Board of Pharmacy to examine and license pharmacists and assistant pharmacists. This was further improved by an amendment passed in 1901.

In 1905, a movement was started to have New Jersey follow the example of her neighboring states in requiring that registered pharmacists must be graduates of reputable schools of pharmacy. The pharmacists of the state had first to be educated to this step, so necessary for professional advancement, and so it was not until 1911 that the New Jersey Pharmaceutical Association approved a draft of a bill for an amendment to the pharmacy act of 1901 the most important provision of which was a section requiring prerequisite education and graduation from a school of pharmacy for the licensed pharmacists.

Since 1912, the legislature of New Jersey has been continuously appealed to for such an amendment to the pharmacy practice act. The Assembly readily responded and on several occasions passed the pharmacists' bill, but the Senate was obdurate and adverse interests

"pigeon-holed" the bill in committee. In 1916, the Committee on Legislation of the State Pharmaceutical Association had the prerequisite amendment introduced first in the Senate and were successful in passing the bill almost unanimously through both houses, but their victory became a defeat when the Governor withheld his approval because it deviated from a policy that he had announced; one of the principal reasons he assigned was the provision for a per diem of $10.00 to each member of the board for services performed for the board.

This year, the committee reframed the bill so that it covered but little more than the desired qualifications for the personnel of the Board of Pharmacy and the educational requirements for applicants for registration as pharmacists or as assistant pharmacists. The bill was favorably acted on by the legislature and was promptly signed by the Governor. The Act provides that every person applying for registration as a pharmacist "shall have been duly graduated from a school of pharmacy complying with the rules and regulations of 'The Board of Pharmacy of the State of New Jersey,' and shall present to the board a certificate from the Commissioner of Education of this State showing that such applicant, before entering the school of pharmacy, had obtained an education equivalent to the standard established by the Board of Pharmacy of the State of New Jersey." Thus leaving to the discretion of the board, the standard of preliminary education to be required and such advancement thereof as, from time to time, they deem wise.

This review of the campaign of education that was necessary to secure needed legislation for the advancement of professional pharmacy in a state that was among the first to enact a pharmacy practice act, is given for the purpose of illustrating the necessity for persistence in the efforts of pharmacists in such matters. It further demonstrates the proverbial slowness of reforms. At least a quarter of a century has passed since the leaders in pharmaceutical education commenced their agitation that students in pharmacy should be required to have sufficient preliminary education to grasp the college studies and that a collegiate education in the sciences associated with the practice of the art of the apothecary should be required by the law of each state. After the lapse of all of these years, we can only state that, with the accessions of this year, eleven or twelve states have adopted laws to that effect. In most of the

states no such legal enactment has as yet been considered and in some of the states that are supporting state universities with pharmacy departments, the door to the practice of pharmacy has been left wide open through its pharmacy law, by permitting the registration of pharmacists without any collegiate education.

Before pharmacy can claim the general recognition as a profession that is so greatly desired, its followers must be professionally educated and the nation-wide adoption of a requirement that every licensed pharmacist must be a graduate of a school of pharmacy is an initial step that is essential for the professional elevation of pharmacy. The stigma of three fourths of our states without such prerequisite laws must be wiped out.

This duty will undoubtedly devolve upon pharmacists themselves and should claim the immediate and persistent attention of every thoughtful person associated with the calling either as a teacher, writer, or practical druggist. Something more than resolutions is needed. A year of active, energetic effort on this movement will accomplish more for the real benefit of pharmacy than a century spent in resoluting. This is an opportunity that the American Pharmaceutical Association has had for years of performing a really great service in the line of its professed work in behalf of the betterment of pharmacy and a duty that it should forthwith enter upon with an active committee of its most capable members. When the educational qualifications of pharmacists are once established, then the objections in official and medical circles to the recognition of pharmacists as professional gentlemen must fall and many of the reforms that we are so strenuously advocating, such as the establishing of a pharmaceutical corps in the Army and Navy, will be more readily attained. As pharmacists it behooves us to appreciate fully the importance of nation-wide prerequisite legislation for pharmacy.

G. M. B.

## Consistent Conservation of Drugs.

From the very beginning of the war, in the summer of 1914, the drug and chemical markets have been in an unsettled condition and, at times, the conditions regarding certain important commodities included in these classifications could be termed as chaotic. The closing of the usual sources of supply, the demands of foreign countries

that prior to this had not looked to America for such supplies, the shortage of labor, the increased cost of production and the uncertainty of transportation were economic problems that the experienced merchant could calculate against but the influence of the enormous speculation engendered by the war made it difficult for conservative business men and the manufacturers to study the market fluctuations and to control the effect of these on their merchandise.

The enormous demand for chemicals for home consumption and for export has been met with the usual American energy. The ability shown, the capital invested and the progress already made demonstrate that many of the chemical industries have been so firmly established that, after this war, under normal trade conditions, we will in these be independent of foreign producers.

The same cannot, however, be said of drugs of vegetable origin and the chemicals derived from such. Here too frequently other problems than the need for energy and capital intervene. Very few plants thrive indiscriminately; most require special soil and climatic conditions. While the cultivation of a few of the continuously scarce and high-priced drugs may be made profitable as industries, those who have given careful study to the subject and, some who from war necessity are now engaged in medicinal plant cultivation, are convinced that in normal times the cost of cultivation with the economic conditions existing in this country will preclude the possibility of successful competition with plants growing naturally or by cultivation in foreign countries where the cost of collection is minimized by the utilization of a class of labor that we do not have in this country. However, we can accomplish much more than has so far been done by continuous scientific study of the possibilities of finding soil, climatic conditions and labor suitable to the needs of the individual plants and moreover the systematic development of methods for the collection and utilization of the vast stores of indigenous and escaped medicinal plants already growing in the United States. This is a much needed field of study for conservation of national resources.

In order to conserve the Nation's resources of both men and materials, Congress by the enactment of "war measures" has placed under executive control all supplies of foods, fuels and other essentials for the national security and defense, as well as transportation

and other public utilities. It becomes the duty of every patriotic citizen to uphold the administration and to coöperate in this important work of conservation in every practicable way. It is doubtful if the majority of the inhabitants of the United States are yet fully alive to the gravity of the situation and the necessity for intensified energy along every line and for consistent conservation of all of our resources.

From the very beginning of the war, the thoughtful reader was impressed by the efforts being put forth by the European nations engaged in warfare to conserve their medical supplies. Especially in the pharmaceutical literature of the central powers, then available, was noted the frequency with which the word "ersatz" was used and the numerous substitutes suggested for drugs and vehicles commonly used for medicines. In Great Britain the need for conserving certain food products and war materials, was considered of sufficient importance to justify the modification of a number of formulas of the British Pharmacopœia, and the suspension of others.

The importance of providing ample supplies of medicines for the needs alike of the government forces and for the civilian population seems to have been largely overlooked. Despite the publicity given to the growing scarcity and enormous advances in the prices of some of the most important medicaments, the national authorities have not made any survey of the needs of the nation for medicines nor of the available supplies in the country. Surveys of fuel, foods, metals, certain chemical products and agricultural needs, etc., have from time to time been made. The schedule of food products accompanying the queries submitted to dealers in these commodities covered more than 100 different foods and presumably the information gained was, at least in part, the basis for the food conservation promulgations.

That a real shortage in many drugs already exists is apparent and this is prone to become more pronounced as the war continues. Competent members of the drug trade should be empowered at once to make a thorough investigation of the medicinal needs of the nation, the supplies in hand and to advise the administration as to the best methods of conserving supplies and obtaining additional or what alternatives may be possible for some articles that may be no longer procurable.

Following the example of the English pharmacists, several com-

munications have appeared in the pharmaceutical journals advocating substitutes for alcohol, glycerin and sugar directed as solvents and vehicles in formulas of the U. S. Pharmacopœia and the National Formulary. While appreciating the spirit actuating the propositions, we cannot believe that the conditions in the United States either now or in the future are likely to be comparable with those existing in England regardng the substances named. The amount of alcohol, glycerin and sugar used in medicines is but a very small portion of that produced and this bears but a small ratio in comparison with the amount consumed in other industries of far less importance to human welfare. Consequently, based upon relative importance, the formulas of the official preparations should be altered only for improvements or in case of such absolute necessity for conserving the basic solvents as we trust never will exist.

The formulas of the U. S. P. and the N. F. are based upon many experiments and in some cases tests of therapeutic activity and permanency of preparation extending over years and before advocating changes in these we should be very certain that the alternatives proposed will yield not preparations appearing similar in viscosity, color or taste but that these will be of equal stability and therapeutic activity. The reduction of the amount of alcohol below what has been determined by such careful experimentation as necessary for exhaustion of a drug or the preservation of its preparations will result in less stable and efficient products. The substitution of diluted commercial glucose for syrup made from cane sugar is likewise questionable as the proneness of glucose solutions to undergo fermentation is well unkown and moreover it is doubtful if such a change would prove to be acceptable to consumers or any real economy at the present value of starch and materials made therefrom. There can be no economy in unstable formulas with the attending waste and danger of contamination.

We are impressed with the fact that many of the suggestions made *have not been tried out practically and appear to be largely academic.* Wherein is the economy in using "powdered glucose" or "saccharin" in place of sugar? Consistent conservation must consider the possibility of obtaining the substitute as a generally distributed article of merchandise and that its use would be a real economy.

We are not prepared to believe that tragacanth paste or irish moss jelly flavored by the addition of chloroform are acceptable

substitutes for the official syrup nor that such alternatives will appeal to the users as proper bases for syrup of hydriodic acid and other official syrups for which these have been suggested. We voice our opinion as opposed to pharmacopœial camouflage.

Another serious objection to many of the suggested "war formulas" is that they offer very broad hints and methods of adulteration which may be continued by the unscrupulous in times of peace. The standards of quality and purity laid down in our official authorities should not be lightly dealt with nor should the bars of the pure drug laws be unnecessarily let down.

A very proper way to effect a sane conservation would be for the physicians wherever possible to simplify their prescriptions. Instead of prescribing fluid extracts of buchu, cascara, sagrada, senna, digitalis, etc., direct infusions of such drugs as those that yield their constituents to water. Vinegar of squill in diminished quantity could replace the syrup and at times solution of iron chloride in place of the tincture and very frequently syrup could with advantage take the place of glycerin and an aromatic water replace an elixir, or one of the weaker alcoholic basic elixirs like almond or vanilla elixir be prescribed in place of the more alcoholic and expensive aromatic elixir. The examples of simplified prescribing, which would lead to consistent conservation without the sacrifice of therapeutic action, could be multiplied many times over and this moreover is a plan in which medicine and pharmacy could work together in a common cause.

G. M. B.

## The Significance of Military Rank.

Rank in the Army and Navy means authority, and authority properly controlled means efficiency. The conferring of military rank upon medical men in the Army and Navy, as proposed by the Owen bill, is vitally necessary for the health and life of the men in the service. Not that physicians will not do their full duty without such rank, but with such distinction they can command sufficient authority to do better work.

Why should rank be granted to other than military officers? After the hideous experiences of former wars and the exigencies arising in military operations in which the welfare of the personnel was not only often jeopardized but sacrificed, the medical arm of

the service asserted its rights. A struggle arose to secure rank for surgeons on a parity with that of the fighting arm. Finally this was achieved in part, yet grudgingly, and, as yet, inadequately. Now, in all the armies and navies of European and Asiatic nations of the first class, the medical officer is clothed with authority parallel with that of officers in command of armies and ships. Immediately and automatically betterments thereupon arose in the conservation of health and efficiency. Why was this achievement of rank so absolutely essential a measure? This query could be best answered by citing numberless gruesome episodes oftentimes involving wasteful destruction of man-power caused by entirely preventable scourges, occurring in recent wars.

These obstructions to medical precautions, serious enough in themselves, were often aggravated by the intolerable and despotic arrogance of certain blindly prejudiced commanding officers. Space forbids narration of instances but any one old enough can recall the humiliating circumstances occurring in the Spanish-American War of 1898. There holocausts followed successful blocking of the best efforts of surgeons to enforce precautionary agents, e. g., in typhoid fever. Due to these nullifications of medical authority, thousands died and other thousands were invalided for life by a readily preventable disease, whereas only hundreds died of wounds or injuries in battle. Now this old struggle, apparently ended, breaks out again, while striving to render our life-saving and health measures more nearly perfect. Is human life worth saving in even so hazardous an occupation as war? On merely the gross basis of *financial economy* the life of a trained soldier is worth close upon $10,000.

Never was the medical arm of a service so well organized and perfected as it is in the United States Army and in the War *now really just beginning.* It has been demonstrated beyond all doubt that the Medical Department must be invested with adequate authority, not only to demand and enforce precautionary and curative measures agreed upon as essential, but also that the individual medical officer shall be empowered to carry out his rulings, *provided they do not jeopardize the military conduct of the War.*

Rank he must have to secure attention, to hold conferences with other military officers; also to get privileges, powers, even products, supplies and equipment, when and where essential, or of hospital supplies, etc. He must have quantivalent rank with those in com-

mand of military operations, or he is unheeded, much less are his requests granted.

To be sure other considerations than those of mere authority come in here. What is the crux of the objections? It is no longer natural for the military officer to be sensitive, to insist upon his traditional social status as a member of the "fighting arm" over all other branches of the service; the present war is showing a mortality among medical men quite as great if not greater in proportion than among the fighting men. Indeed, he need no longer be chary of conceding place at the military fireside to Doctors, Dentists, Veterinarians, and now Pharmacists. Again, are we a democracy or an aristocracy? Why the Army—why the Navy? To fight the battles of the country or each other? Who does the fighting? The enlisted man? What are the solemn obligations of the Government? To keep the fighting force in the pink of fighting condition, as far as possible, physically and mentally, by giving the medical man of the Army proper authority to enforce his medical judgment, or to have the medical men continue to take their orders in matters medical from non-medical men?

J. Madison Taylor, M.D.

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## A METHOD FOR CLEANING DIGITALIS, WITH A STUDY OF THE INORGANIC CONSTITUENTS.

By C. H. Rogers and E. L. Newcomb.

For many years close observers have noticed the great variation in the cleanliness of different samples of digitalis. Even those who have been interested in the preparation of the drug from cultivated plants have called attention to the fact that an apparently unnecessary excess of foreign matter was often present in the drug produced. With these thoughts in mind an attempt was made in connection with the harvesting and preparation of the 1917 crop of digitalis produced in the Medicinal Plant Garden of the College of Pharmacy of the University of Minnesota to produce a drug with a minimum amount of adhering material.

Collection and Drying.—The seedlings were transferred from the cold frames to the garden during the last of June and the first of July. The plants made an excellent growth and were irrigated

during dry spells. Very few plants died during the season. The harvesting, which was done the latter part of September and the first of October, was practically completed before freezing weather set in, although there had been some slight frost. The total harvest represented approximately 600 Kg. of fresh leaves. The collecting was done largely by the students of the College of Pharmacy. All of the leaves were collected from plants of the first year's growth.

The leaves were collected by grasping a handful and cutting near the ground with a sharp knife, the lower parts of the petioles being left with the base of the plant. The more heavily laminated upper portions of the petioles were for the most part included with the drug collected. Only the rich green fully matured leaves were selected. The average yield of the green leaves, as determined by weighing the product from 500 plants, was 240 grammes per plant.

After collecting the leaves were taken into the Medicinal Plant Laboratory, where as much adhering soil as possible was removed by brushing. On account of the large quantity of drug collected it was impractical to hand brush the entire lot.

The soil in all of the plots in which the digitalis was grown was rather rich in humus admixed with sandy loam. The major portion of the ground was campus ground in lawn the preceding year and was plowed late in the spring. Attention should also be called to the fact that the plantings were for the most part adjoining a dusty and much-used driveway. During dry spells plants near the roadway were frequently covered with a grayish-white dust from the road. A number of rains just prior to collecting apparently removed the majority of the dust.

After brushing, the weighed leaves were spread one layer deep on screens or tin trays and placed in drying ovens which were heated by gas. On account of limited oven space the preliminary drying of part of the crop was carried out by placing the leaves on window screens, and these were then placed in the laboratory; the finishing, however, was done in the ovens. A temperature of approximately 80 degrees Centigrade was maintained until the petioles would break with a sharp fracture. Previous determinations have shown from two to four per cent. of moisture remaining in the leaves at this stage. The dried leaves were then weighed and the percentage of moisture lost determined. The average of numerous determinations showed a loss in weight on drying of between 80 and 82 per cent.

CLEANING PROCESS.—The dried leaves were placed in seven steel storage bins holding approximately 16 Kg. each. The subsequent cleaning and study of the drug was carried on some two months later, at which time it was desired to prepare the drug in a form suitable for the preparation of U. S. P. infusion and tincture. The handling of the dried drug gave evidence of the presence of much adhering and easily separable silt, although the product appeared much cleaner than the average commercial sample and was of a uniform rich green color. The ease with which the adhering material separated suggested the possibility of further cleaning by shaking the broken leaves on a coarse screen.

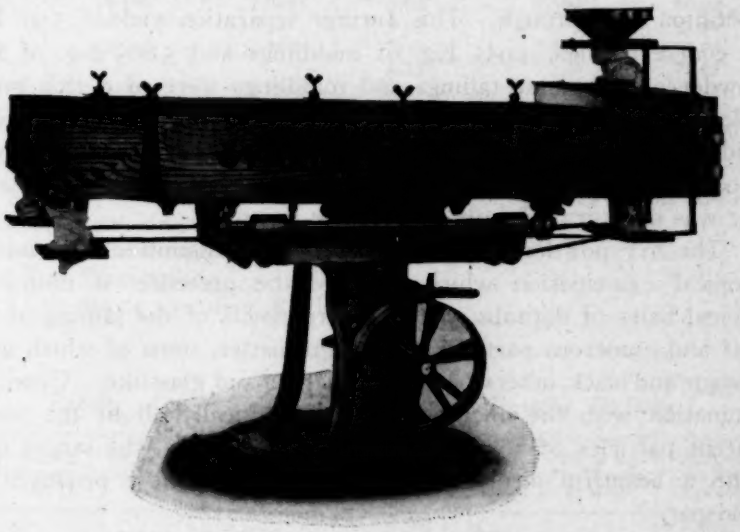

FIG. 1. Schutz-O'Neill Gyrator Sifter, used for special cleaning of digitalis and general drug sifting in the Drug Milling Laboratory, College of Pharmacy, University of Minnesota.

A bronze window screen having fourteen meshes to the linear inch was placed upon the body of a No. 1 Schutz-O'Neill Gyrator Sifter, the top and regular screen frame being first removed. A frame 3½ inches high was constructed around the edges of the window screen and fitted tightly down upon it. Approximately 2 Kg. of digitalis leaves were thrown upon the screen at one time, and then the machine, which was operated by a 2 H. P. motor, was set in motion. This operation resulted in separating 17.128 Kg. of screenings from a total of 81.984 Kg. of clean leaves. These

screenings apparently contained much select drug, and it was deemed an unnecessary waste to discard the same; therefore, they were subjected to the following process:

One of the regular sifting frames supplied with the gyrator sifter was placed on the machine. This frame when used on the machine provides for the separation of the powder, being sifted into three degrees of fineness. Approximately three fifths of the area of the frame used is covered with a 50-mesh silk bolting cloth, one fifth with a 17-mesh linen screen and the remaining area is open for the tailings or coarse portion. The dust-proof top was placed on the machine and the 17.138 Kg. of screenings from the previous operation run through. This further separation yielded 5.97 Kg. of coarse tailings, 4.955 Kg. of middlings and 5.805 Kg. of fine powder. The coarse tailings and middlings were of a rich green color and apparently free from adhering soil. The tailings and middlings were returned to the cleaned drug from the screening process, after samples had been reserved for study. The fine powder was dark gray, faintly tinged with green.

The fine powder above referred to was submitted to a microscopical examination which revealed the presence of numerous typical hairs of digitalis, very few fragments of the lamina of the leaf and numerous particles of foreign matter, some of which were opaque and black, others being transparent and glasslike. Upon examination with the micropolariscope practically all of the transparent particles of foreign matter polarized light, the larger ones with a beautiful display of colors similar to those produced by feldspar.

Ash Determinations.—Ash determinations were made on the original drug before any of the cleaning processes were carried out and subsequently on the various separations effected. A composite sample carefully selected from the original crop yielded on three successive determinations 11.368 per cent., 11.38 per cent., and 11.59 per cent. of ash. While these figures are considerably below the percentage of ash permitted by U. S. P. IX, it was considered advisable to separate all of the foreign matter possible, thus producing a drug of exceptional cleanliness and probably within the two per cent. limit of foreign material.

The results of the separation by the two methods described together with ash determinations of the drug taken from each of the seven bins are given in the following two tables:

TABLE I.

*Results of Screening Process in Cleaning Digitalis. With 14 Mesh Screen.*

| No. of Drug Bin. | Weight in Kg. | Wt. in Kg.† of C. Screen. | Per Cent.* Ash C. Screen. | Wt. in Kg.‡ of F. Screen. | Per Cent. Ash F. Screen |
|---|---|---|---|---|---|
| | | | 9.237 | | 26.64 |
| 1. | 14.856 | 12.246 | 9.26 | 2.610 | 26.659 |
| | | | 9.183 | | 18.9 |
| 2. | 16.554 | 12.019 | 9.24 | 4.535 | 22.33 |
| | | | 9.224 | | 38.00 |
| 3. | 12.026 | 9.525 | 9.62 | 2.501 | 30.00 |
| | | | 9.49 | | 24.85 |
| 4. | 11.565 | 9.071 | 9.466 | 2.494 | 24.88 |
| | | | 9.755 | | 21.29 |
| 5. | 8.389 | 7.029 | 9.62 | 1.360 | 20.94 |
| | | | 8.99 | | 31.92 |
| 6. | 9.524 | 7.210 | 8.73 | 1.814 | 29.15 |
| | | | 9.25 | | 24.63 |
| 7. | 9.070 | 7.256 | 8.91 | 1.814 | 27.27 |
| | Total 81.984 | Total 64.356 | Average 9.284 | Total 17.138 | Average 26.24 |

* Samples were run through a No. 6 Sieve in order to obtain uniform portions for ash determinations and other tests.

† Coarse Screenings.

‡ Fine Screenings.

§ Lost during operation .398 Kg.

TABLE II.

*Results of Gyrator Sifting Process in Cleaning Digitalis.*

| Total Wt. Kg. F. Screenings (See Table I). | Wt. in Kg. Coarse Tailings. | Per Cent. Ash Coarse Tailings. | Wt. in Kg. Middlings, 17 Mesh. | Per Cent. Ash Middlings. | Wt. in Kg. Fine Powder, 50 Mesh. | Per Cent. Ash Fine Powder. |
|---|---|---|---|---|---|---|
| | | 8.92 | | 10.07 | | 57.6 |
| *16.730 | 5.970 | 8.91 | 4.955 | 10.26 | 5.805 | 56.02 |

* 398 Kg. reserved from first operation for sample.

Our knowledge of the chemical and pharmacological activity of foreign inorganic substances in digitalis is limited. It has been suggested by Wilbert[1] that, during the making of tincture or infusion, the clay may adsorb more or less of the therapeutically active constituents of the drug and thus render it less valuable. If such foreign matter were entirely inert a given quantity of such drug would be materially less active than if well cleaned. The normal ash of digitalis is probably between 8 and 10 per cent. Attention

[1] Wilbert, *Bulletin*, Univ. of Wis., Serial 829, page 50.

should be called to the fact that commercial samples have been reported upon which run over 25 per cent. of ash. Such samples of course would not come within the present U. S. P. IX limits. In case the foreign inorganic material adsorbs the therapeutically active constituents it would then seem especially important that all such extraneous matter be discarded.

A study of Table I clearly shows that by a process of screening, digitalis containing over 11 per cent. of ash may be separated into two portions, one yielding an ash content of 9.284 per cent., and the other passing through a screen of fourteen meshes to the linear inch, yielding 26.24 per cent. The second portion represented approximately 20 per cent. of the entire quantity, to discard all of which seemed an unnecessary waste, especially as its ash content indicated that it was about four fifths digitalis.

The second table shows that a further process of separation may be employed to almost completely separate the foreign inorganic material. The fine siftings yielded over 56 per cent. of ash, while the coarser portions were approximately 9 and 10 per cent., respectively. The total weight of fine siftings was 5.805 Kg. or approximately 7 per cent. loss from the original drug. This may seem like a considerable loss, especially to those who handle digitalis in large quantities. The actual loss of digitalis, however, is less than 3½ per cent., the remaining portion being foreign matter.

In view of the unquestionable wide variation in the chemical nature of the foreign material which may be present, it would seem desirable to carry the process of elimination of foreign matter just as far as possible, notwithstanding the fact that it may increase the cost of the drug. The U. S. P. IX definition for digitalis permits not more than two per cent. of foreign matter. The requirement is also made that the ash of digitalis shall not exceed fifteen per cent. In other words, there is an allowance for variation in the normal ash of digitalis up to thirteen per cent. While some may question the advisability of requiring digitalis to be 98 per cent. pure, nevertheless by the application of methods as outlined in this paper such requirement is apparently met with even when the foreign matter consists chiefly of microscopic material.

Special Digitoxin Tests.—Qualitative chemical tests for the presence of digitoxin were made on the portions separated in the cleaning process. The method employed in conducting these tests

was as follows: Ten mils of a 10 per cent. infusion of digitalis leaves were placed in a separatory funnel, ten mils of chloroform added and the two liquids shaken. The mixture was allowed to settle and then 10 mils of a mixture of equal parts of 95 per cent. alcohol and ether were added. The chloroform-ether mixture was filtered off and evaporated. The residue was dissolved in 3 mils of acetic acid. To this solution one drop of a 10 per cent. solution of ferric chloride was added and then a layer of sulphuric acid was introduced at the lower portion of the test tube, by means of a pipette. A brown red color changing to a bluish green at the zone of contact is indicative of digitoxin (the digitoxin zone).

The results of the application of the test when applied to each of the separated portions are given in the following table:

TABLE III.

*Digitoxin Tests.*

| Petioles. (See Table No. 5.) | Coarse Screenings. (Table No. 1.) | Coarse Tailings. (Table No. 2.) | Middlings. (Table No. 2.) | Fine Powder. (Table No. 2.) |
|---|---|---|---|---|
| Faint | Positive | Positive | Positive | Negative |

It will be noted that all samples gave a positive reaction for digitoxin with the exception of the fine siftings which yielded 56 per cent. of ash and which on microscopic examination revealed practically no tissues of digitalis leaf except the trichomes. These tests on the siftings are particularly interesting in that they indicate that the hairs of digitalis are devoid of digitoxin. The elimination then of siftings in this case would in no way be detrimental to the drug unless other medicinally valuable glucosides were present. Whether or not these fine siftings have a detrimental effect upon the quality of tinctures or infusions prepared from the drug will be reported upon in a subsequent paper.

Attention is here called to what is apparently an error in considering the green color developing in the above so called digitoxin test as digitoxin. Digitoxin is insoluble in water and hence cannot possibly be present in the infusion unless solution is effected by the presence of other substances in the leaf. Our extensive work with Digitalis during the past six years has uncovered much evidence which leads us to believe that digitoxin does not exist as such in the leaf of the growing digitalis plant. The immediate drying by artificial heat of the freshly collected digitalis leaves undoubtedly les-

sens the formation of decomposition products one of which may be digitoxin. It is our opinion that the green color zone in the above test is due to digitalein rather than digitoxin.

THE DISCARDING OF FINE PORTIONS.—The question of the advisability of discarding the finer portions of digitalis during the process of grinding may be determined only by taking into consideration a number of factors. In any event, the drug should be

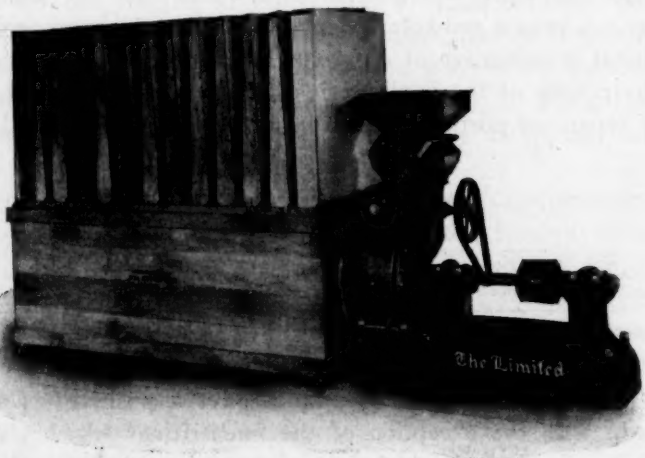


FIG. 2. Schutz-O'Neill Limited Pulverizer, used for the preparation of uniform finely powdered digitalis and other drugs. One of seven mills in the Drug Milling Laboratory, College of Pharmacy, University of Minnesota.

cleaned as thoroughly as possible before grinding and the nature and ash content of the finer portion of the grind should be determined before it is eliminated. By the use of the Schutz-O'Neill Limited Pulverizer "set fine" and run at a speed of 3,300 R. P. M. or faster, a thoroughly dried digitalis leaf may be reduced at one operation to such a degree of fineness that 75 per cent. or more will pass through an 80-mesh bolting cloth at the first sifting, a large proportion of the powder being finer than a number 80. In such powder much of the drug may be in fine particles of the same size as adhering inorganic matter and obviously it would be a mistake to throw away the finer parts of such a portion. Grinding in this manner is advisable for the preparation of digitalis to be dispensed in one-grain capsules on physicians' prescriptions. On the other hand, a coarse grind of digitalis may yield a relatively

small proportion of number 80 powder which may run high in ash, depending upon the thoroughness of the cleaning process and the character of the soil in which the plant is grown, *i. e.*, fine clay or coarse sandy humus.

In this connection it is interesting to note that many of the ash determinations reported in the literature for digitalis and based upon examination of drug prepared from wild-growing plants represent low ash figures. It is reasonable to believe that wild-growing plants well mulched with roadside grass, leaves or other organic material would be protected to a greater degree from inorganic contamination than field-grown plants under intensive cultivation.

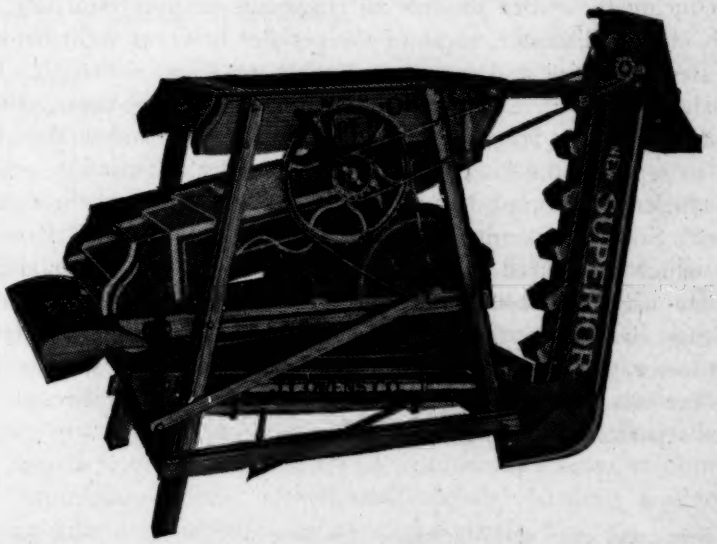


FIG. 3. Owens Superior Fanning Mill, used for special preparation of digitalis for infusion, the cleaning of drug seeds, fruits and other purposes, Drug Milling Laboratory, College of Pharmacy, University of Minnesota.

PREPARATION OF DIGITALIS FOR INFUSION.—A portion of the cleaned digitalis leaves was to be used for the manufacture of the official infusion. The leaves were to be packed in number 10 one-ounce-capacity gelatin capsules, each containing 3.75 grammes, sufficient to prepare 250 mils of infusion, the usual amount prescribed. The U. S. P. directs that bruised digitalis be used for the preparation of the infusion. An attempt to bruise the properly prepared digitalis, produced in Minnesota, resulted in the immediate production of a very fine powder due to the fact that the drug contains

very little moisture, is chiefly parenchymatous and is therefore easily pulverized. A fine powder is unquestionably not desired for the preparation of the infusion. It would be an improvement if the U. S. P. formula directed the use of digitalis in number 6 powder. This would insure a uniformity in the degree of fineness which is not likely to be obtained when using a variety of samples and following the present directions.

In order to reduce the Minnesota digitalis to a proper degree of comminution for the preparation of the infusion the following process was employed. The cleaned leaves were run through a sieve having six meshes to the linear inch. This operation resulted in reducing the entire product to fragments of approximately one sixth inch in diameter, some of the petioles however went through the sieve endwise and were two inches or more in length. Obviously it would have been difficult to have packed the capsules with these long petiole fragments present. The next problem therefore was to separate the long fragments of petioles. This was quickly and efficiently accomplished by running the broken drug through an Owen's Superior Fanning Mill which was driven by a 2 H. P. motor and which was fitted with screens as follows: The upper tier of screens used represented a "zinc wheat gang" of four frames (having circular perforations one fifth of an inch in diameter). The lower sand frame used was a regular 30 × 30 wire screen. The fan was shut off entirely. The drug was then run through and three separations obtained as follows:

TABLE IV.

| | | |
|---|---|---|
| Select and quite uniform number six drug ..... | 7.029 | Kg. |
| Petiole fragments, 1 to 8 cm. in length with practically no lamina present ..... | 1.6 | Kg. |
| No. 30 passing thru the lower screen..... | loss, 375. | Gm. |

Total ash determinations were made of the three separations, which gave the following results:

TABLE V.

| | Select Portion. | Petiole Fragments. | No. 30 Powder. |
|---|---|---|---|
| *A*..... | 8.85 | 8.78 | 12.81 |
| *B*..... | 8.97 | 8.75 | 13.97 |
| Average ..... | 8.91 | 8.76 | 13.3 |

It will be noted that the coarse lamina and petiole portions, both of which were practically free from adhering matter, gave nearly an identical amount of ash. This is probably explained by the fact that they both consist primarily of parenchyma tissue. The finer portion shows an increase in the per cent. of ash as was the case in the previous cleaning process.

Some of the separated petiole fragments were tested for digitoxin by the method previously described. The result, which is recorded in Table III, it will be noted, was not more than a very faint trace. In view of the large amount of mucilaginous material present in the petioles we feel that this statement should be confirmed by physiological methods.

THE CHEMICAL EXAMINATION OF THE ASH.—On account of the possible effect of foreign inorganic material in the digitalis, it is highly important that the chemical composition of the same be known. Qualitative and quantitative examinations were therefore made of the ashes obtained by incinerating the portions separated by the cleaning processes.

Qualitative determinations were made on (*A*) ash of digitalis cleaned by screening, (*B*) ash of digitalis middlings, (*C*) ash of digitalis siftings, (*D*) ash of fine powder from digitalis siftings. Positive reactions were obtained from each of the above samples for iron, aluminum, manganese, calcium, magnesium, potassium, sodium, silicon and carbonates. Negative results were obtained for lead, mercury, silver, bismuth, copper, cadmium, arsenic, strontium, tin, chromium, cerium, nickel, zinc, cobalt, barium, antimony, lithium, chlorides, sulphates, nitrates, phosphates.

The presence or absence of manganese in digitalis has been the subject of a number of investigations. Our results tend to indicate that in Minnesota digitalis it is present only in small amounts. This confirms the earlier results obtained by one of us (Newcomb[2]), also by Burmann.[3] Manganese was positively identified in the ash of the clean digitalis leaves by the following tests:

(*a*) In neutral solution by ammonium sulphide—flesh-colored precipitate of MnS.

(*b*) Nitric acid solution with lead peroxide—violet-colored solution.

(*c*) Heating on platinum foil with KOH and potassium nitrate—green color.

[2] Newcomb, AMER. JOUR. PHARM., Vol. 84, pp. 201–214.

[3] Burmann, *Schweiz. Wochschr.*, 1911, p. 562.

(*d*) Heating on platinum foil with KOH and potassium chlorate—green color, changed to purple of permanganates upon boiling with water.

Samples of soil from the plots from which the digitalis was grown were collected, incinerated and tested for the presence of manganese by methods as given above. The results were positive.

Particular attention should be called to the method employed in conducting tests "*c*" and "*d*." When a considerable quantity of manganese is present in the ash, as in Stafford Allen and Sons, English digitalis, or in digitalis from the state of Washington, only a small amount of KOH and potassium chlorate is necessary to produce the characteristic green color, however, when the amount of manganese is extremely small as in Minnesota digitalis, it is necessary to use larger quantities of these reagents, in order to observe the reaction satisfactorily.

The test for potassium also indicates that it is a normal constituent of the leaf. On account of the excessive cost of reagent potash was not run quantitatively.

PERCENTAGE OF ASH INSOLUBLE IN HCL.—The following table clearly indicates that the 9.284 per cent. of ash obtained from the clean digitalis by screening contained but very little foreign matter, as only 4.35 per cent. of the ash was insoluble in hydrochloric acid. On the other hand nearly two thirds of the screenings ash represented material insoluble in HCl. This quality of the ash is even

TABLE VI.

*HCl Insoluble Ash in Digitalis.*

| Material. | Determination. | Percentage Ash Average. | Percentage of Ash Insol. in HCl. | Percentage of Ash Insol. in HCl—Average. |
|---|---|---|---|---|
| Digitalis cleaned by screening..... | 1 | 9.284* | 4.47 | 4.35 |
| | 2 | | 4.23 | |
| Digitalis screenings..... | 1 | 26.24* | 60.45 | 61.65 |
| | 2 | | 62.86 | |
| Digitalis middlings and siftings..... | 1 | 8.915† | 10.20 | 9.41 |
| | 2 | 10.16† | 8.63 | |
| Fine powder from digitalis siftings..... | 1 | 56.81 | 70.98 | 70.99 |
| | 2 | | 71.00 | |

* Average of 14 samples.

† No. 1, Tailings; No. 2, Middlings. Samples mixed for insol. ash determinations.

more pronounced in the second table which shows that 71 per cent. of the fine powder ash was insoluble in HCl. The tables illustrate that there is a direct relation between the percentage of HCl insoluble ash and the amount of inorganic foreign matter in the drug.

QUANTITATIVE ASH DETERMINATIONS.—The following table shows the amount of elements present in the ash, calculated as oxides, and the alkalinity representing chiefly carbonates. It will be noted that the high percentage of ash caused by foreign matter was due chiefly to silica with some aluminum. The high percentage of alkalinity in the ash of the clean drug as compared to that of the siftings would indicate the combination of iron, calcium, magnesium, potassium and sodium with organic acids in the normal leaf, and the relatively small amount of these salts in the ash of the siftings. The small amount of these salts in the ash of the siftings, the low alkalinity together with the dual capacity in which some of these elements occur make it difficult if not impossible to correctly proportionate the constituents.

TABLE VII.

*Quantitative Table of Inorganic Constituents of Digitalis.*

| Ash of | Determination. | Total Siliceous Material. | Si as $SiO_2$. | Iron as $Fe_2O_3$. | Al as $Al_2O_3$. | Ca as CaO. | Mg as MgO. | K and Na as $K_2O$–$Na_2O$. | Mils N/10 $H_2SO_4$ per Gm. Ash M. O. Ind.* |
|---|---|---|---|---|---|---|---|---|---|
| Digitalis cleaned by screen..... | *A* | 11.58 | 10.84 | 9.59 | 3.62 | 20.10 | 23.93 | 31.18† | 42.56 |
| | *B* | 11.53 | 10.67 | 9.52 | 3.51 | 20.03 | 23.53 | 31.88† | 42.59 |
| | Av. | 11.55 | 10.76 | 9.55 | 3.56 | 20.06 | 23.73 | 31.53 | 42.575 |
| Fine powder from siftings ..... | *A* | 73.18 | 44.75 | 5.04 | 3.33 | 9.45 | 9.77 | | 8.08 |
| | *B* | 73.41 | 45.16 | 5.32 | 3.46 | 9.21 | 9.15 | | 8.12 |
| | Av. | 73.29 | 44.96 | 5.18 | 3.39 | 9.33 | 9.46 | | 8.10 |

SUMMARY.—Foreign inorganic material adhering to dry digitalis leaves may be easily separated by a mechanical process.

By the application of such process supplies of digitalis may be made much more uniform with respect to adhering material.

The evident variation in the chemical nature of foreign material

* Methyl orange used as indicator.

† By difference.

which may be present would seem to demand that the process of elimination be carried just as far as practical.

The small amounts of digitalis lost in the processes of cleaning are not sufficient to give satisfactory reactions for digitoxin by qualitative chemical tests.

The digitalis lost in the cleaning process consists chiefly of the hairs of the leaf which apparently contain no digitoxin.

The advisability of discarding the finer portions of digitalis during the process of grinding is dependent upon the nature of the foreign matter and the method of milling employed. All digitalis should be well cleaned before being milled.

The total ash of leaves from cultivated plants usually averages higher than the ash of leaves from wild-growing plants.

The rapid increase in the supplies of digitalis from cultivated plants suggests the necessity of giving special attention to the cleaning process.

The U. S. P. requirement that digitalis for infusion be bruised does not insure uniformity in degree of fineness of drug, which different pharmacists use in making infusions. It would be advisable if the U. S. P. formula directed the use of digitalis in number 6 powder for the same. A mechanical process may be employed to reduce the drug to a uniform number 6 powder. The petioles are deficient in digitoxin, appear to contain much mucilaginous material and may largely be separated in producing a uniform number 6 powder.

Gelatin capsules afford an excellent means for packing digitalis to be used in making infusions.

The total percentage ash of clean petioles of digitalis is approximately the same as the clean lamina portions.

Manganese appears to be a constant constituent of digitalis leaves but varies in amount in samples obtained from different sources.

Siliceous foreign matter insoluble in HCl may be almost entirely separated by a cleaning process.

Digitalis produced in Minnesota normally contains iron, calcium, magnesium, manganese, potassium and sodium.

The small percentage of aluminum occurring as a silicate in the Minnesota digitalis would indicate that the therapeutic activity of preparations made from this drug would not be seriously impaired by the adsorptive properties of the silicate.

# TINCTURE OF GINGER U. S. P.

By J. P. Snyder,

Norwich, N. Y.

The U. S. P. directs that tincture of ginger shall be made from Jamaica ginger and percolated with 95 per cent. alcohol and that a tincture of ginger so made shall meet the following requirements:

Evaporate 10 Gm. of tincture of ginger to dryness in a tared dish on the water-bath; the yield does not exceed 2 per cent. When treated with 20 mils of cold distilled water, not more than 15 per cent. of this residue dissolves. It contains about 90 per cent. of alcohol and in addition to these requirements a qualitative test is given by which capsicum or other pungent substitutes are detected. When one carefully examines the above standards, he finds that the only one which is really worth while and which attains its object, of preventing adulterated tincture finding its way upon the market, is the alcohol requirement of about 90 per cent. In fact, the statement that it shall not yield more than 2 per cent. of solids in reality invites adulteration by the unscrupulous. For it has definitely been shown by several different investigators that the average total solids of tincture of ginger is in the neighborhood of 1.5 per cent. and, at present, with the U. S. P. standard of not more than 2 per cent. solids, there is nothing that would prevent one from adding to the average tincture of ginger percolated with 95 per cent., once or twice its volume of straight alcohol, and still have a product that would meet the Pharmacopœia requirements, although in reality it would only be one half strength or less. In fact, it is possible to have a strictly U. S. P. preparation that would fail to meet the standard of the Food and Drug Department, which is, or at least has been, that tincture of ginger shall contain in each 100 Cc. the alcohol-soluble of 20 Gm. of ginger.

The U. S. P. requirement, that not more than 15 per cent. of the solids is soluble in 20 mils of water, has given rise to considerable discussion, as it seems possible to prepare a tincture of ginger from strictly prime Jamaica ginger root that fully meets the U. S. P. requirements and find that more than 15 per cent. of the solids are soluble in 20 Cc. of water. This condition may, however, be brought about by lack of definite directions in the Pharmacopœia

for obtaining the water-soluble solids While the U. S. P. states the amount of water with which the solids are to be treated, it does not state the length of time which the water is to remain in contact with the solids and whether it is to be 5, 10, or 15 minutes, 1 hour, 2 hours, or three hours or longer, is left entirely to the discretion of the analyst, and as the amount of water-soluble solids will vary with the length of time the water is left in contact with the solids, it should be definitely stated how long this should be. From the fact that, after adding water to the solids, a sticky gummy mass results, the average chemist would probably leave it in contact with the water for, at least, a couple of hours, and rub it frequently with a rubber-tipped rod.

Since we have been finding that several of the tinctures of ginger have yielded by our interpretation of the water-soluble method of the U. S. P. results higher than 15 per cent., it has occurred that it might be advisable to consult available literature upon the subject, prepare samples and make a general investigation.

Street and Morrison, Bulletin 137, U. S. P. Dept. of Agriculture, give the following method for determining water-soluble solids: "Add 15 to 20 Cc. of water, at room temperature, to the residues obtained in the total solids determination. Stir with a glass rod at frequent intervals during the three hours. Because of the gummy character of many residues, it is sometimes difficult to secure contact with the solvent. Wash into a 50 Cc. flask with water and make up to the mark. Filter this solution through a dry plaited filter and evaporate an aliquot of 25 Cc. and dry to constant weight in a water oven."

The U. S. P. excludes this method of Street & Morrison, when it specifies only 20 Cc. of water, for, of course, the residue could not then be washed with sufficient water to bring it up to 50 Cc.

Street and Morrison prepared one sample of tincture of ginger and determined the total solids to be 1.43 per cent., 14.7 per cent. of which was soluble in water by the method given above. From the results of this one tincture of ginger they concluded that properly prepared tincture of ginger should show a specific gravity of about 0.82 and contain, at least, 93 per cent. of alcohol by volume, from one or two per cent. of solids, practically all of which is soluble in alcohol, and not over 15 per cent. soluble in cold water.

Street and Morrison, in drawing conclusions from one sample of tincture of ginger, have at least very little scientific data to base their

claims upon, and in placing the limit for water-soluble solids at 15 per cent., when they found 14.7 per cent., they have adopted an extremely narrow limit. When it is taken into consideration that they are using a method which will show variations greater than 0.3 per cent. in the hands of different operators, as they themselves state in their method, that "because of the gummy character of many of the residues, it is sometimes difficult to secure proper contact with the solvent."

In the same article, we read the following: "34 samples sold as flavoring extracts and 14 sold as tincture of ginger were examined, but only the analysis of certain ones which are typical and a general summary of all samples will be given. Seven of the extracts were classed as standard composition and seven as legally labeled compounds and twenty as below standard, or adulterated. *Of the tinctures, 12 are of standard quality.* As the tinctures are of special interest to us an examination of the analytical data accompany this article is advisable, from which we find that the average analysis of these 12 standard tinctures is as follows:

| | |
|---|---|
| Specific gravity | 0.8371 |
| Alcohol by volume | 93.33 |
| Total solids | 1.38 |
| Soluble in alcohol | 1.30 |
| Soluble in water | 0.23 |
| Per cent. of the total solids soluble in alcohol | 96 |
| Per cent. of the total solids soluble in water | 17 |

In one paragraph we read that they conclude, from one sample of tincture of ginger prepared by them, that not over 15 per cent. of the solids should be soluble in water, and, in another, that the average of the water-soluble of 12 standard tinctures is 17 per cent. This, surely, is not consistent, to state that the water-soluble should be less than 15 per cent. and then to consider as standard quality 12 tinctures, the average water-soluble of which is 17 per cent.

Lythgoe and Nurenburg, *Journal of Industrial and Engineering Chemistry*, December, 1911, prepared a sample of tincture of ginger, using 200 Gm. of Jamaica ginger to the liter and percolated with 95 per cent. alcohol. They present the following analysis:

| | Per Cent. |
|---|---|
| Alcohol by volume | 88.76 |
| Total solids | 1.40 |
| Soluble in alcohol | 1.33 |

| | |
|---|---|
| Water-soluble | 0.24 |
| Per cent. of total solids soluble in alcohol | 95 |
| Per cent. of total solids soluble in water | 18 |

In addition to this the authors also examined 4 tinctures of ginger, which they consider to be strictly U. S. P., and the following are part of the results which they obtained:

| | Alc. by Vol. | Total Solids. | Alc. Soluble, Per Cent. | Water Sol. | Per Cent. of Solids Sol. in | |
|---|---|---|---|---|---|---|
| | | | | | Alc. | Water. |
| 1 | 91.64 | 1.23 | 1.09 | 0.10 | 88 | 8.1 |
| 2 | 88.84 | 1.26 | 1.16 | 0.19 | 94 | 15.1 |
| 3 | 88.04 | 1.59 | 1.38 | 0.27 | 86 | 17.0 |
| 4 | 78.92 | 1.48 | 1.47 | 0.24 | 99 | 16.2 |

Here are 4 tinctures of ginger, one of which was prepared by the authors themselves, which yields a per cent. of solids soluble in water, greater than 15, and are considered strictly U. S. P. Of course they were examining these tinctures according to the U. S. P. VIII, where no standards were given for tinctures of ginger, but there is no reason to presume that tincture of ginger, made at that time, using the same quantity of Jamaica ginger and the same alcoholic menstruum, should not yield results similar to those of the tinctures prepared at this time by the U. S. P. IX method.

Harrison and Sullivan, Association of Official Agriculture Chemists, give the results of analysis of about 25 tinctures of ginger, many of which they prepared themselves, and from their results concluded that tincture of ginger U. S. P. should comply with the following requirements. Specific gravity at 25° C. not above 0.827. Alcohol by volume not less than 90 per cent. Non-volatile matter from 1.25 to 1.75 per cent. They did not even find it necessary to determine the water-soluble portion of the solids and accordingly have given no requirements in the standards which they propose.

A sample of tincture of ginger was prepared in our laboratory from Jamaica ginger, which met the botanical description of the U. S. P. and gave the following results when examined chemically:

| | | |
|---|---|---|
| Alcohol-soluble | 6.45 per cent. | U. S. P. plus 4 per cent. |
| Non-volatile, ether-sol. | 4.47 per cent. | U. S. P. plus 2 per cent. |
| Water-soluble | 10 per cent. | U. S. P. plus 8 per cent. |
| Ash | 3.7 per cent. | U. S. P. plus 8 per cent. |

These results would indicate that the Jamaica ginger met the U. S. P. requirements in every particular. The alcohol used in the percolating of the ginger contained 94.9 per cent. by volume of absolute alcohol. The specific gravity of the tincture made from this ginger was 0.8173 and it contained 88.5 per cent. of alcohol by volume. For the determination of the water-soluble solids, 4 samples of approximately 10 Gm. each were evaporated to dryness on the steam bath and dried in desiccator. The U. S. P. directs that 10 Gm. be used, but as it is out of the question to weigh accurately exactly 10 Gm. of a volatile substance like tincture of ginger, about 10 Gm. were taken in each case. The following are the results obtained for total solids from these four samples:

1. 1.80 per cent.
2. 1.77 per cent.
3. 1.81 per cent.
4. 1.85 per cent.

In addition two other samples were evaporated to dryness on the steam bath and then dried in the oven to constant weight at 100° C. and gave the following results:

5. 1.74 per cent.
6. 1.76 per cent.

To sample No. 1 20 Cc. of water were added and the solids thoroughly rubbed with a rubber-tipped glass rod to bring the sticky mass of solids in contact with the water. The water, after remaining on the ginger solids for five minutes was removed, filtered through 9 Cm. filter and the first two or three Cc. rejected.

Sample No. 2 was treated in the same manner as No. 1 with the exception that the water was allowed to remain in contact with the solids for 15 minutes, No. 3 for 1 hour and 30 minutes and No. 4 for 2 hours and 30 minutes. 10 Cc. of the filtrates, from each of the samples were transferred to a tared evaporating dish and evaporated to dryness, dried to constant weight at 100° C. and the following water-soluble percentages obtained.

1. 16.7 per cent.
2. 18.8 per cent.
3. 22.9 per cent.
4. 21.5 per cent.

## Conclusions.

From the above it would appear that tincture of ginger, the percentage of water-soluble solids of which are greater than 15 per cent., can be prepared from strictly U. S. P. Jamaica ginger, using 95 per cent. alcohol as the menstruum. This is confirmed by Lythgoe and Nurenburg and, in a way, by Street and Morrison who, although they consider that tincture of ginger should yield less than 15 per cent. of water-soluble solids, find that 12 tinctures of ginger are of standard quality, in which the average yield of solids soluble in water is 17 per cent.

The per cent. of water-soluble solids is variable, depending upon the length of time the water is allowed to remain in contact with the solids and also, to a great extent, upon the manner in which the solids are brought in contact with the water. If the water-soluble solids requirement serves any practical purpose, and it is to be retained, the U. S. P. should state the way and manner in which it is to be obtained, and also establish a limit that can be met when the tinctures are prepared from the U. S. P. Jamaica ginger and 95 per cent. alcohol.

In determining the total solids, the statement in the Pharmacopœia should be changed from "Evaporate 10 Gm. of tincture of ginger" to "Evaporate about 10 Gm. of tincture of ginger accurately weighed," as the tincture is volatile and it is impractical and unnecessary to weigh exactly 10 Gm. Also it would probably be better to state that the total solids should be dried to constant weight at 100° C., as somewhat lower results may be obtained and also more accurate checks if the determination is made in this manner. *The minimum limit as well as the maximum should be stated for the total solids to prevent adulteration by dilution.* The following standards in addition to qualitative tests for capsicum or other pungent substitute would probably be all that would be necessary to prevent adulteration of tincture of ginger and at the same time show the product to be of a good quality.

Alcohol about ..... 90 per cent. by volume.
Specific gravity about ..... 0.82
Non-volatile solids, not more than 1.75, nor less than 1.25 per cent.

Analytical Laboratories,
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# METHODS OF GAS WARFARE.[1]

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(Communicated by L. J. Briggs.)

All I can do in the short time available is to give you, if I can, a general idea of what gas warfare really means on the Western Front at the present time. Some of you may have gotten the idea that gas is just an incident, and that there is not as much attention being paid to it as there was two years ago. That idea is entirely wrong. Gas is used to a tremendous extent, and the amount that has been and is being hurled back and forth in shells and clouds is almost unbelievable. I will try to give you a general idea of what is occurring and make the lecture rather a popular than a technical description. I shall also, for obvious reasons, have to confine myself to describing what the Germans have been doing, and will say nothing about what we are doing.

Possibly the best plan would be to state more or less chronologically what occurred. I happened to be present at the first gas attack and saw the whole gas business from the beginning. The first attack was made in April, 1915. A deserter had come into the Ypres salient a week before the attack was made, and had told us the whole story. They were preparing to poison us with gas, and had cylinders installed in their trenches. No one believed him at all, and no notice was taken of it.

Then came the first gas attack, and the whole course of the war changed. That first attack, of course, was made against men who were entirely unprepared—absolutely unprotected. You have read quite as much about the actual attack and the battle as I could tell you, but the accounts are still remarkably meager. The fellows who could have told most about it didn't come back. The Germans have claimed that we had 6,000 killed and as many taken prisoners. They left a battlefield such as had never been seen before in warfare, ancient or modern, and one that has had no compeer in the whole war except on the Russian front.

What the Germans expected to accomplish by it I am not sure.

[1] Report of a lecture delivered before the Washington Academy of Sciences on January 17, 1918. Reprinted from *Journal of the Washington Academy of Sciences.*

Presumably they intended to win the war, and they might conceivably have won it then and there if they had foreseen the tremendous effect of the attack. It is certain that they expected no immediate retaliation, as they had provided no protection for their own men. They made a clear and unobstructed gap in the lines, which was only closed by the Canadians, who rallied on the left and advanced, in part through the gas cloud itself.

The method first used by the Germans, and retained ever since, is fairly simple, but requires great preparation beforehand. A hole is dug in the bottom of the trench close underneath the parapet, and a gas cylinder is buried in the hole. It is an ordinary cylinder, like that used for oxygen or hydrogen. It is then covered first with a quilt of moss, containing potassium carbonate solution, and then with sand bags. When the attack is to be made the sand bags and protecting cover are taken off the cylinder, and each cylinder is connected with a lead pipe which is bent over the top of the parapet. A sand bag is laid on the nozzle to prevent the back "kick" of the outrushing gas from throwing the pipe back into the trench. Our own methods are practically identical with those first used by the Germans.

The success of a cloud gas attack depends on thorough preparation beforehand. The attackers must know the country, the layout of the trenches, and the direction and velocity of the wind with certainty. Favorable conditions are limited practically to wind velocities between twelve and four miles an hour. A wind of more than twelve miles an hour disperses the gas cloud very rapidly. An upward current of air is the worst foe of gas. The weight of the gas is not an important factor in carrying it along, for it mixes rapidly with air to form the moving "cloud." The time occupied by a gas attack is too short to permit of much diffusion of the gas out of the original mixture.

The gas attack must be planned very carefully. If the trench line is very irregular it is likely that gas will flow into a portion of one's own trenches. The limits of safety in wind direction are thus determined by the direction of the lines of the trenches. The Germans use a 40° angle of safety; that means that on a given straight portion of the front the wind direction must lie between the two directions which make angles of 40° with the neighboring sections of the front. The most suitable type of country is where the ground slopes gently away from where the gas is being dis-

charged. The Germans made one mistake in believing that hilly or wooded country would not do. This was refuted by the French, who made a successful gas attack in hilly and wooded country in the Vosges, as admitted in a captured German report. If the country is flat like that about Ypres, and the wind direction is right, there is very little difficulty about making an attack, especially if the enemy does not know anything about it. The element of surprise is important.

German gas attacks are made by two regiments of Pioneers, with highly technical officers, including engineers, meteorologists, and chemists. They brought their first cylinders into the line without our knowing anything about it, except from the deserter's report, which was not believed. The element of surprise was greatly lessened when we began to know what to look for and to recognize the sounds incident to the preparation of a gas attack.

The first attack was made with chlorine. If a gas attack is to be made with gas clouds, the number of gases available is limited. The gas must be easily compressible, easily made in large quantities, and should be considerably heavier than air. If to this is added the necessity of its being very toxic and of low chemical reactivity, the choice is practically reduced to two gases: chlorine and phosgene. Chlorine is to gas warfare what nitric acid is to high explosives. Pure chlorine did not satisfy quite all the requirements, as it is very active chemically and therefore easily absorbed. Many men in the first attack who had sufficient presence of mind saved themselves by burying their faces in the earth, or by stuffing their mufflers in their mouths and wrapping them around their faces.

There were several gas attacks of almost exactly the same kind early in 1915. There was no gas between the end of May, 1915, and December, 1915, and by that time adequate protection had been provided.

The first protection was primitive. It consisted largely of respirators made by women in England in response to an appeal by Kitchener. They were pads of cotton wool wrapped in muslin and soaked in solutions of sodium carbonate and thiosulphate; sometimes they were soaked only in water. A new type appeared almost every week. One simple type consisted of a pad of cotton waste wrapped up in muslin together with a separate wad of cotton waste. These were kept in boxes in the trenches, and on the word "gas" six or eight men would make a dive for the box, stuff some waste

into their mouths, then fasten on the paid and stuff the waste into the space around the nose and mouth. But this got unpopular after a bit, when it was discovered that the same bits of waste were not always used by the same men. During the early part of 1915 this was the only protection used.

Then came the helmet made of a flannel bag soaked in thiosulphate and carbonate, with a mica window in it. A modified form of this device with different chemicals is still used in the British army as a reserve protection. It is put over the head and tucked into the jacket, and is fool-proof as long as well tucked down. This stood up very well against chlorine.

In 1915 we got word from our Intelligence Department of a striking kind. It consisted of notes of some very secret lectures given in Germany to a number of the senior officers. These lectures detailed materials to be used, and one of them was phosgene, a gas which is very insidious and difficult to protect against. We had to hurry up to find protection against it. The outcome was a helmet saturated with sodium phenate. The concentration of gases when used in a cloud is small, and 1 to 1,000 by volume is relatively very strong. The helmet easily gave protection against phosgene at a normal concentration of 1 part in 10,000. That helmet was used when the next attack came in Flanders, on the 19th of December. This attack was in many ways an entirely new departure and marked a new era in gas warfare.

There are three things that really matter in gas warfare, and these were all emphasized in the attack of December. They are: (1) increased concentration; (2) surprise in tactics; (3) the use of unexpected new materials.

Continued efforts have been made on both sides to increase the concentration. The first gas attack, in April, 1915, lasted about one and a half hours. The attack in May lasted three hours. The attack in December was over in thirty minutes. Thus, assuming the number of cylinders to be the same (one cylinder for every meter of front in which they were operating), the last attack realized just three times the concentration of the first, and six times the concentration obtained in May. Other cloud gas attacks followed, and the time was steadily reduced; the last attacks gave only ten to fifteen minutes for each discharge. We believe that the cylinders are now put in at the rate of three for every two meters of front, and may even be double banked.

The element of surprise came in an attack by night. The meteorological conditions are much better at night than during the day. The best two hours out of the twenty-four, when steady and downward currents exist, are the hour between sunset and dark and the hour between dawn and sunrise. Gas attacks have therefore been frequently made just in the gloaming or early morning, between lights. This took away one of the easy methods of spotting gas, that of seeing it, and we had to depend upon the hissing noises made by the escaping gas, and upon the sense of smell.

Another element of surprise was the sending out of more than one cloud in an attack. After the first cloud the men would think it was all over, but ten minutes or half an hour later there would come another cloud on exactly the same front. These tactics were very successful in at least one case, namely, the attack near Hulluch in 1916. Some of the troops discarded their helmets after the first wave and were caught on the second, which was very much stronger than the first.

Efforts were also made to effect surprise by silencing the gas. But silencers reduced the rate of escape so greatly that the loss of efficiency from low concentration more than made up for the gain in suddenness. Another method was to mix the gas up with smoke, or to alternate gas and smoke, so that it would be difficult to tell where the gas began and the smoke ended.

The last attack made on the British by this means was in August, 1916. Since that time the Germans have used gas three times on the West Front against the French, and have also used it against the Italians and the Russians. It has been practically given up against the British, although the method is by no means dead.

The last attack was a slight set-back in the progress of gas defense. The casualties had been brought down to a minimum, and, as shown by the fact that the percentage of deaths was high, protection was complete in all cases where used, casualties being due to unpreparedness in some form. The attack in question was brought on under difficult conditions for the defenders, as it was made on new troops during a relief when twice as many men were in the trenches as normally. Furthermore, they had to wear helmets while carrying their complete outfit for the relief. This was the second time the Germans caught us in a relief, whether through information or luck we cannot say.

The protection that had been devised against phosgene proved

effective at the time, but provision was made to meet increased concentration of phosgene. We never had any actual evidence during the attack that phosgene was being used, as no samples were actually taken from the cloud, but cylinders of phosgene were captured later. Glass vacuum tubes, about 10 by 30 Cm., with a tip that could be broken off and then closed by a plasticine-lined glass cap, were distributed, but the only one that came back was an unopened tube found in a hedge, and marked by the finder "Dangerous; may contain cholera germs." In a gas attack everybody keeps quiet or else has a job on hand, and conditions are not conducive to the taking of gas samples. The original types of vacuum tube were smaller than those now used.

There was a long search for materials that would absorb phosgene, as there are few substances that react readily with it. The successful suggestion came from Russia. The substance now used very extensively by all is hexamethylenetetramine (urotropine) $(CH_2)_6N_4$, which reacts very rapidly with phosgene. Used in conjunction with sodium phenate, it will protect against phosgene at a concentration of 1:1,000 for a considerable period. An excess of sodium hydroxide is used with the sodium phenate, and a valve is provided in the helmet for the escape of exhaled air. The valve was originally devised so that the hydroxide would not be too rapidly carbonated, but it was found in addition that there is a great difference in ease of breathing and comfort if a valve is placed in the mask. The helmet is put on over the head, grasped with left hand around the neck and tucked into the jacket. This form is still used in reserve.

By this time gas shells were beginning to be used in large numbers, and it became evident that protection by a fabric could not be depended on with certainty. The box type of respirator was the next development. Respirators have to fulfill two requirements which are quite opposed to one another. In the first place they should be sufficiently large and elaborate to give full protection against any concentration of any gas, whereas military exigency requires that they be light and comfortable. It is necessary to strike a balance between these two. Upon a proper balance depends the usefulness of the respirator. Oxygen apparatus will not do on account of its weight and its limited life. Two hours' life is excessive for that type. The side that can first force the other to use oxygen respirators for protection has probably won the war.

The concentrations of gas usually met with are really very low. As has been said, a high concentration for a gas cloud is 1 part in 1,000, whereas concentrations of two or three per cent. can be met by respirators depending on chemical reactivity. One such respirator is a box of chemicals connected by a flexible tube with a face-piece fitting around the contours of the face, and provided with a mouthpiece and nosepiece.

As regards the chemicals used there is no secret, for the Germans have many of the same things. Active absorbent charcoal is one of the main reliances, and is another suggestion that we owe to the Russians. Wood charcoal was used in one of their devices and was effective, but most of the Russian soldiers had no protection at all.

We wanted to protect against chlorine, acids and acid-forming gases, phosgene, etc., and at one time were fearful of meeting large quantities of hydrocyanic (prussic) acid (HCN). At one period every prisoner taken talked about the use of prussic acid, saying that the Kaiser had decided to end the war and had given permission to use prussic acid. Protection was evidently needed against it. The three things that then seemed most important were: (1) chlorine and phosgene; (2) prussic acid; (3) lachrymators. Charcoal and alkaline permanganate will protect against nearly everything used, even up to concentrations of ten per cent. for short periods.

The German apparatus, developed about the same time, is of different pattern, and is still employed. It consists of a small drum, attached directly to the front of the face-piece, and weighs less than the British respirator but must be changed more frequently. It has no mouthpiece. The chemicals are in three layers: first an inside layer of pumice with hexamethylenetetramine, in the middle a layer of charcoal (sometimes blood charcoal), and outside baked earth soaked in potassium carbonate solution and coated with fine powdered charcoal.

As regards the future of the gas cloud, it may be looked upon as almost finished. There are so many conditions that have to be fulfilled in connection with it that its use is limited. It is very unlikely that the enemy will be able to spring another complete surprise with a gas cloud.

The case is different with gas shells. The gas shells are the most important of all methods of using gas on the Western Front,

and are still in course of development. The enemy started using them soon after the first cloud attack. He began with the celebrated "tear" shells. A concentration of one part in a million of some of these lachrymators makes the eyes water severely. The original tear shells contained almost pure xylyl bromide or benzyl bromide, made by brominating the higher fractions of coal-tar distillates.

The German did his bromination rather badly. As you know, it should be done very carefully or much dibromide is produced, which is solid and inactive. Some of the shells contained as much as twenty per cent. dibromide, enough to make the liquid pasty and inactive. The shells used contain a lead lining, and have a partition across the shoulder, above which comes the T. N. T. and the fuse. These shells had little effect on the British, but one attack on the French, accompanied by a very heavy bombardment with tear shells, put them out badly. The eyes of the men were affected, and many of the men were even anesthetized by the gas, and were taken prisoner.

Our first big experience was an attack at Vermelles. The Germans put down a heavy barrage of these shells and made an infantry attack. The concentration was great, the gas went through the helmets, and the men even vomited inside their helmets. But it is difficult to put down a gas barrage, and there is danger that it will not be a technical success. In the instance cited certain roads were not cut off sufficiently, so that reinforcements got up. This attack, however, opened our eyes to the fact that, as in the case of gas clouds, concentration would be developed so as to make it high enough to produce the required effect under any circumstances.

When the Germans started using highly poisonous shells, which was at the Somme in 1916, they did not attend to this sufficiently, although enormous numbers of shell were used. The substance used was trichloromethyl-chloroformate, but not in great strength. It had no decided reaction on the eyes, hence the men were often caught.

The quantity of gas that can be sent over in shells is small. The average weight in a shell is not more than six pounds, whereas the German gas cylinders contain forty pounds of gas. To put over the same amount of gas as with gas clouds, say in five minutes per thousand yards of front, would require a prohibitive number of guns and shells. It becomes necessary to put the shells on definite

targets, and this, fortunately, the Germans did not realize at the Somme, although they have found it out since.

The use of gas out of a projectile has a number of advantages over its use in a gas cloud. First, it is not so dependent on the wind. Again, the gunners have their ordinary job of shelling, and there is no such elaborate and unwelcome organization to put into the front trenches as is necessary for the cloud. Third, the targets are picked with all the accuracy of artillery fire. Fourth, the gas shells succeed with targets that are not accessible to high explosives or to gas clouds. Take, for instance, a field howitzer, dug into a pit with a certain amount of overhead cover for the men, who come in from behind the gun. The men are safe from splinters, and only a direct hit will put the gun out of action. But the gas will go in where the shell would not. It is certain to gas some of the men inside the emplacement. The crew of the gun must go on firing with gas masks on, and with depleted numbers. Thus it nearly puts the gun out of commission, reducing the number of shots say from two rounds a minute to a round in two minutes, and may even silence it entirely. Another example is a position on a hillside with dugouts at the back, just over the crest, or with a sunken road behind the slope. Almost absolute protection is afforded by the dugouts. The French tried three times to take such a position after preparation with high explosives, and each assault failed. Then they tried gas shells, and succeeded. The gas flows rapidly into such a dugout, especially if it has two or more doors.

Among the effective materials used by the Germans for gas shells were mono- and tri-chloromethyl-chloroformate. Prussic acid never appeared; the Germans rate it lower than phosgene in toxicity, and the reports concerning it were obviously meant merely to produce fear and distract the provisions for protection.

During the last five months the actual materials and the tactics used by the Germans have undergone a complete change. The lachrymator shells are less depended upon than formerly for "neutralization," but are still a source of annoyance. Mere annoyance, however, may be an effective method of neutralizing infantry. For instance, where large amounts of supplies and ammunition are being brought up there are always cross-roads where there is confusion and interference of traffic. A few gas shells placed there make every man put on his mask, and if it is a dark night and the roads are muddy, the resulting confusion can be only faintly imagined.

It may thus be possible to neutralize a part of the infantry by cutting down their supplies and ammunition.

The use of a gas shell to force a man to put on his mask is practically neutralization. If at the same time you can hurt him, so much the better. Hence the change in gas-shell tactics, which consists in replacing the purely lachrymatory substance by one that is also poisonous.

One substance used for this method of simultaneously harassing and seriously injuring was dichloro-diethysulphide (mustard gas). Its use was begun in July of last year at Ypres, and it was largely used again at Nieuport and Armentieres. A heavy bombardment of mustard-gas shells of all calibers was put on these towns, as many as 50,000 shells being fired in one night. The effects of mustard gas are those of a "super-lachrymator." It has a distinctive smell, rather like garlic than mustard. It has no immediate effect on the eyes, beyond a slight irritation. After several hours the eyes begin to swell and inflame and practically blister, causing intense pain, the nose discharges freely, and severe coughing and even vomiting ensue. Direct contact with the spray causes severe blistering of the skin, and the concentrated vapor penetrates through the clothing. The respirators of course do not protect against this blistering. The cases that went to the hospitals, however, were generally eye or lung cases, and blistering alone took back very few men. Many casualties were caused by the habit that some of the men had fallen into of letting the upper part of the mask hang down so as not to interfere with seeing. The Germans scored heavily in the use of this gas at first. It was another example of the element of surprise in using a new substance that produces new and unusual symptoms in the victims.

Up to the present time there has been no material brought out on either side that can be depended on to go through the other fellow's respirator. The casualties are due to surprise or to lack of training in the use of masks. The mask must be put on and adjusted within six seconds, which requires a considerable amount of preliminary training, if it is to be done under field conditions.

Among other surprises on the part of the Germans were phenylcarbylamine chloride, a lachrymator, and diphenylchloroarsine, or "sneezing gas." The latter is mixed in with high explosive shells or with other gas shells, or with shrapnel. It was intended to make a man sneeze so badly that when he puts on his mask he is not able

to keep it on. The sneezing gas has, however, not been a very great success.

All bombardments now are of this mixed character. The shells used are marked with differently colored crosses, and definite programs are laid down for the use of the artillerymen.

As regards the future of gas shells, it should be emphasized that the "gas shell" is not necessarily a gas shell at all, but a liquid or solid shell, and it opens up the whole sphere of organic chemistry to be drawn upon for materials. The material placed inside the shell is transformed into vapor or fine droplets by the explosion and a proper adjustment between the bursting charge and the poisonous substance is necessary. Both sides are busy trying to find something that the others have not used, and both are trying to find a "colorless, odorless, and invisible" gas that is highly poisonous. It is within the realm of possibilities that the war will be finished, literally, in the chemical laboratory.

The Germans have not altered their type of respirator for some time, and it is not now equal in efficiency to the British or American respirator. The German respirator, even in its latest form, will break down at a concentration of 0.3 per cent. of certain substances. The German design has given more weight to military exigency, as against perfect protection, than has the British. Another thing that weighs against changes in design is the fact that the German, already handicapped by the lack of certain materials, must manufacture 40,000,000 respirators a year in order to supply his Austrian, Bulgarian, and Turkish allies, as well as his own army.

In the British and American armies the respirator must always be carried with the equipment when within twelve miles of the front. Between twelve and five miles a man may remove the respirator box in order to sleep, but within five miles he must wear it constantly. Within two miles it must be worn constantly in the "alert" position (slung and tied in front). When the alarm is given he must get the respirator on within six seconds. The American respirator is identical with the British. The French have a fabric mask made in several layers, the inner provided with a nickel salt to stop HCN, then a layer with hexamethylenetetramine; it has no valve and is hot to wear. The French also use a box respirator, consisting of a metal box slung on the back, with a tube connecting to the face mask; the latter is of good Para rubber and is provided with a valve. One disadvantage of this form is the danger of tearing

the single rubber sheet. The German mask now contains no rubber except one washer; the elastic consist of springs inside a fabric, and the mask itself is of leather. It hardens and cracks after being wet, and is too dependent upon being well fitted to the face when made.

(The lecturer exhibited various types of gas shells, helmets, masks, and respirators.)

The following compounds have been used by the Germans in gas clouds or in shells:

1. Allyl-iso-thiocyanate (Allyl mustard oil), $C_3H_5NCS$ (shell).
2. Benzyl bromide, $C_6H_5CH_2Br$ (shell).
3. Bromo-acetone, $CH_2Br \cdot CO \cdot CH_3$ (hand grenades).
4. Bromated methyl-ethyl-ketone (bromo-ketone), $CH_2Br$-$COC_2H_5$ or $CH_3 \cdot CO \cdot CHBr \cdot CH_3$ (shell). Dibromo-ketone, $CH_3$-$COCHBr \cdot CH_2Br$ (shell).
5. Bromine, $Br_2$ (hand grenades).
6. Chloro-acetone, $CH_2Cl \cdot COCH_3$ (hand grenades).
7. Chlorine, $Cl_2$ (cloud).
8. Chloromethyl-chloroformate (Palite), $ClCOOCH_2Cl$ (shell).
9. Nitro-trichloro-methane (chloropicrin or nitrochloroform), $CCl_3NO_2$ (shell).
10. Chlorosulphonic acid, $SO_3 \cdot H \cdot Cl$ (hand grenades and "smoke pots").
11. Dichloro-diethylsulphide (mustard gas), $(CH_2ClCH_2)_2S$ (shell).
12. Dimethyl sulphate, $(CH_3)_2SO_4$ (hand grenades).
13. Diphenyl-chloro-arsine, $(C_6H_5)_2AsCl$ (shell).
14. Dichloromethyl ether, $(CH_2Cl)_2O$ (shell).
15. Methyl-chlorosulphonate, $CH_3ClSO_3$ (hand grenades).
16. Phenyl-carbylamine chloride, $C_6H_5NCCl_2$ (shell).
17. Phosgene (carbonyl chloride), $COCl_2$ (cloud and shell).
18. Sulphur trioxide, $SO_3$ (hand grenades and shell).
19. Trichloromethyl-chloroformate (diphosgene, superpalite), $ClCOOCCl_3$ (shell).
20. Xylyl bromide (tolyl bromide), $CH_3C_6H_4CH_2Br$ (shell).

# THE MANGANESE CONTENT OF THE ASH OF CERTAIN DRUGS.[1]

By L. E. Westman and R. M. Rowat, Ottawa, Canada.

Although the significance of the presence of manganese in plants and its relation to plant metabolism is not yet clear, the quantity of manganese observed to be present in the barks of certain laxative drugs and in other portions of plants used for similar purposes was considered by the authors to be worthy of general mention.

Hafner and Krist have pointed out in a qualitative way that a large number of drug plants contain manganese. They were able to obtain positive tests for manganese in 164 drugs listed in the Austrian Pharmacopœia, and were led to this investigation by the occurrence of a greenish color in the ash of many drugs. This color is formed when the ash is strongly heated in a muffle from the manganite salts present and is certainly most marked in many instances, especially those where an extract of the drug has been evaporated and ashed. This is due to the fact that only a small fraction of the lime salts present in plants is extracted by methods of percolation, and in the relative absence of these salts the manganese that is extracted becomes observable in the ash.

The method adopted by these authors in their tests was as follows: 3 to 4 Gm. samples of the drug were ashed and the ash dissolved in 8 to 10 Cc. of dilute nitric acid (free from hydrochloric acid), filtered if necessary, then heated, and after the addition of a small amount of brown, manganese-free lead peroxide, was brought to boiling. When manganese was present the liquid acquired a more or less intense purple-red color according to the amount. This reaction naturally fails in the presence of chlorides.

According to Pichard, the seeds of plants are quite rich in manganese, and Jadin and Astruc find that the aërial portions of plants contain more manganese than the subterranean. McHargue tabulates 51 determinations made on nuts, shells, seeds, and seed coatings, obtaining for his two highest results 0.04 per cent. manganese in dried potato peelings, and 0.09 per cent. in dried turnip tops. Manganese has been shown by Headden to be present in all samples of wheat examined in amounts from 0.004 to 0.007 per cent.

[1] From *The Journal of the American Chemical Society.*

## Results and Discussion.

The results obtained are tabulated below and are calculated on the dry basis at 110°:

Table I.

| Drug Examined. | Per Cent. of Manganese Present. |
|---|---|
| Rhamnus Frangula (or alder buckthorn bark) | 0.0242 to 0.0674 ± 0.0003 |
| Cinnamomum Cassia Cortex (or cassia bark) | 0.0624 ± 0.0003 |
| Rhamnus Purshiana (cascara sagrada) | 0.0137 to 0.0223 ± 0.0003 |
| Podophyllin (root) | 0.0052 ± 0.0002 |
| Senna (leaves) | 0.0043 ± 0.0002 |
| Rhubarb (root) | 0.0036 ± 0.0002 |
| Rhamnus Californica (mature bark) | 0.0033 ± 0.0002 |
| Licorice (root) | 0.0026 ± 0.0001 |
| Jalap (root) | 0.0024 ± 0.0001 |
| Euonymus Atropurpureus (wahoo bark) | 0.0021 ± 0.0001 |
| Cassia pulp | 0.0017 ± 0.0001 |
| Aloes (barbadoes) | 0.0006 ± 0.00005 |

From data available it would appear that both Rhamnus Frangula and cassia bark show a higher content of manganese than any other similar plant tissue previously examined. It was found that all samples of these barks did not show the same manganese content, and observed limits of the amounts present are given above. The lower numbers represent mature or thick bark, while the higher numbers were obtained from thinner bark. It is quite possible that an examination of specially selected bark would widen this range, but the work reported was carried out on various samples and it is thought that it covers the range fairly well. Factors involving the change of manganese content with the season have not, however, been studied. In an attempt to locate the cells or region of the bark giving the highest content of manganese, both inside and outside scrapings were examined. It was found for Rhamnus Purshiana that the concentration of manganese in the inner third of the bark was about double that present in the outer third. This may indeed be generally true and would go to show that although manganese is present in the outer layers of the bark it really functions more particularly in the inner layers where plant metabolism is more actively going on. The accidental presence of definite amounts of manganese in these barks cannot be accepted as an explanation, while the conception that a definite concentration of manganese is a factor in the normal metabolism of any particular species seems more probable. It is natural to expect that a plant using a higher con-

centration of manganese in certain working cells might leave behind in the older bark a higher uniform residue. In any case it seems evident that definite amounts of manganese may be found present in all or any of the different parts of plants.

### Manganese Fraction Soluble in Water.

By methods of percolation, and by boiling the powdered barks in water, it was found that about one quarter of the total manganese present may be extracted from members of the Rhamnaceæ family by this means.

Table II.

| Bark Examined. | Total Manganese in the Bark, Per Cent. | Manganese Extracted, Per Cent. |
|---|---|---|
| Rhamnus Frangula | 0.0242 | 0.0058 |
| Rhamnus Purshiana | 0.0137 | 0.0029 |
| Rhamnus Californica | 0.0033 | 0.0008 |

The numbers are based on dry samples as above and are the average results of several good duplicate determinations, showing the uniformity with which manganese is extracted from such materials. Manganese thus extracted does not appear to have the properties of inorganic manganese and is not removed from these solutions by ordinary means. This at once leads to a new method for distinguishing between these closely related species and it follows from this that the development of a "Manganese Number" for commercial extracts of such drugs would seem to be a quite reasonable and practical method of procedure in their examination. It is our hope to develop such an application in the near future.

*Method of Estimation.*—The analytical work reported was accomplished by a simple application of the ammonium persulphate method for the determination of small amounts of manganese. The work was done in two ways, depending on the manganese found present by initial trials.

Wherever 10 Gm. samples are available, the following procedure may be recommended: If the presence of manganese is known to be of the order of 0.015 per cent. or higher, it is possible to titrate directly the permanganate formed with standard sodium arsenite solution. If the percentage is lower than this it is more feasible to compare the color developed with solutions of known permanganate content which have been carefully prepared and correspond

with the unknown solution with regard to acidity and general ion concentration.

In cases of the first order of Manganese content, 10 Gm. samples of the well-ground materials were ashed in platinum in a muffle and 20 Cc. of pure concentrated sulphuric acid was added. The sample was then heated till the acid fumed freely in order to remove chlorides. It was then cooled and diluted with distilled water, washed out into a 500 Cc. beaker and diluted to at least 300 Cc. To this solution was added one Cc. of a silver nitrate solution (5 Gm. in 100 Cc.) and the whole warmed to about 80°. Approximately one Gm. of ammonium persulphate was gradually stirred into this solution, which was then heated on the steam bath as long as the color of the permanganate deepened. By check analyses the reaction was found generally to be complete at the end of 30 minutes. The solution was, however, heated 45 minutes and allowed to cool. When cold it was rapidly titrated with sodium arsenite solution which had been standardized against known amounts of permanganate developed in the same way. Unless the dilution is sufficient a brown hydrated form of manganese will separate out after the addition of the ammonium persulphate. Where the percentage of manganese is as high as 0.06 a volume of 500 Cc. is necessary when working on a 10 G. sample.

In cases where the manganese was of a lower order than 0.01 per cent. the color was developed as above from a 10 Gm. sample in a 200 Cc. volumetric flask and compared by means of a colorimeter with standard solutions of permanganate.

Our thanks are due to Dr. J. M. Francis, of the Parke Davis Co., Detroit, Mich., for kindly supplying us with genuine samples of some of the above materials.

*Summary.*—(1) The manganese content of certain laxative drugs has been measured along with allied species of plants.

(2) It has been found that for the family Rhamnaceæ the manganese is extracted in water proportionally to its total amount in the bark.

(3) A practical use of this determination is suggested leading to the establishment of a "Manganese Number" for extracts of these drugs.

# THE USE OF BACTERIA IN THE PREPARATION OF NATURAL INDIGO.[1]

We have had occasion to refer to the promising work now in progress in India, to establish upon a genuine scientific basis the treatment of the indigo plant, for the transformation of the indican present into indigo. The advance thus far made by those in charge of this important investigation, Mr. Hutchinson, the bacteriologist, and Mr. Davies, the chemist, is outlined in a recent bulletin of the Pusa Research Institute, and is here briefly summarized:

The possibility of bacterial action was suggested by three facts. Firstly, the gas produced during the process of fermentation in the steeping vat varies in quantity and composition; secondly, chemical decomposition commences near the walls of the vat, and then spreads inwards, this suggesting that the presence of bacteria on the walls has something to do with the change; and, thirdly, there is a curious divergence in the yield, not only between different factories, but between vats of the same factory worked under apparently identical conditions, and in particular, when a new vat is brought into use it takes some days for it to reach the normal yield of the factory.

These observations suggested that the theory of transformation so far accepted, by which the main credit for the change was given to a constituent in the plant itself, was incomplete. The conversion of indican, the crude form of the dye, into indoxyl—which is subsequently converted into indigo by oxidation—appeared to depend in part on outside agencies, and laboratory researches showed this to be the case. It has now been established that bacterial action plays a considerable part in the process of change, being in fact essential to the extraction of the maximum yield of indigo. This is a discovery of great potential importance, for it at once supplies an explanation of, and promises a remedy to, the extraordinary differences in yield which occur in adjacent factories. In Bihar, for example, the variations are from one to several hundred per cent., and no satisfactory reason for their occurrence was previously advanced. It now appears to be certain that the high-yielding vats are equipped with a full stock of beneficial bacteria, while the low yielders are either inadequately stocked with these or contain actively hostile bacteria. Mr. Hutchinson has isolated the adverse

[1] From *The Chemical Engineer.*

bacteria, and also obtained pure cultures of several of the beneficent species, obtaining exact measurements of their indoxyl-producing powers. They exhibit considerable variation.

The introduction and propagation of friendly species can be effected in several ways. The steeping vats can be inoculated with pure cultures, or the multiplication of the beneficial organisms already present can be fostered by the provision of conditions favorable to their growth. Mr. Hutchinson believes that a combination of these two methods will prove the most satisfactory. At the same time the bacteria must be given fuller scope for their action. Being restricted to the walls and floor of the vat, their area of operations is limited, and if the vats are too large their productive powers fail to act on the more remote portions of the indican solution. It therefore appears desirable to adapt the vats so as to increase the ratio of wall surface to cubic contents. What the most efficient ratio is has still to be determined, but this is mainly a matter of experiment. It will certainly vary from factory to factory until the bacterial conditions are uniform. Again, the character of the wall surface is a factor of importance. A smooth cement wall is probably not the ideal surface, in which case a substitute has to be found. This also should soon be revealed by experiment. Once discovered, the practical benefits would be considerable, and quickly repay the producers for the necessary alterations.

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## ANDALUSIAN ESSENTIAL OILS.[1]

By Louis G. Dreyfus, Jr., Consul, Malaga, Spain.

The essential-oil industry is said to have been established in Malaga about twenty years ago. At first the production was on a small scale, but it has steadily increased; especially so since the outbreak of the European war, when the German production of cheaper extracts and essences was shut off.

There are two modern steam distilleries at Malaga, with experienced chemists in charge of the works; one of these is a German firm and the other is Belgian. Another steam distillery is located at Jaen. There are also hundreds of small producers scattered throughout Malaga, Jaen, and Granada Provinces, with direct-fire

[1] From *Commerce Reports.*

stills holding from 100 to 500 pounds of plant. The product of these small establishments is not so good as that of the steam distilleries. Furthermore, instead of three fourths of a pound of essential oil from 100 pounds of plant, less than one half pound is recovered in these country stills.

Many of the country producers are furnished with stills by the large essential-oil dealers under an arrangement whereby the product turned out by them is to be sold, at previously contracted prices, exclusively to the dealer who supplied the still.

*Spain Said to Lead World in Production.*—While there are no definite statistics available on this subject Spain is said to lead the world in the production of spike, rosemary, thyme, sage, pennyroyal, and the finest quality of geranium rose oil. Although practically no herbs or plants for the production of essential oils are cultivated, raw materials are readily obtainable in the mountains, and the distilleries are able to operate all through the year. The full season for sage is May and June; for thyme and rosemary, July and August; for fennel, spike, and lavender, September and October. During the winter, when the plants are scarcest, the large distilleries devote themselves to redistillation.

In this part of Spain the actual plant or its leaves and not the flowers are distilled in making essential oils. The probable reason for this is that the air is so charged with salt that the perfume of the flowers is destroyed.

Spanish essential oils are used in the preparation of perfumery, soap, medicine, spirits, and for the adulteration of other essential oils and spirits. Spike is used to adulterate the much finer lavender. The local juniper is employed in the adulteration of the finer French, Belgian, and Dutch juniper. An internal-revenue tax is levied upon distilled anise. It is said that oil of fennel, upon which there is no tax, is used to adulterate the anise. Geranium rose oil is used in the adulteration of rose oil.

*Gathering the Plants.*—Men, women, and children scour the mountains to cut the plants from which the essential oils are made. The plants are gathered into bunches, loaded onto the backs of donkeys, and brought directly to the place selected for the distillation. The lavender plant is too fragile to be transported any great distance and must be distilled where it actually grows. Consequently, if any of the factories desire to produce lavender oil, they must send a portable still to the scene of operations. In the laven-

der season the Sociedad Santiago sends a small steam still, with a capacity of 100 gallons, operated by a traction engine, into the country.

The prices paid for the various plants and berries to be used for distillation vary considerably. The present price of most of them ranges from 1.20 to 1.80 pesetas per 100 pounds delivered at the place of distillation. Cultivated herbs, such as the geranium, bring a higher price. Juniper berries cost 12 pesetas per 100 pounds.

*The Process of Manufacture.*—In Andalusia the aromatic plants are not dried before distillation, but are used as they come from the country. They are placed in the distillery vats; steam is then introduced from the botton, so that it diffuses evenly and forces upward through the plants; the heat of the steam expands the globules of oil which are contained in the minute cells of the leaves, causing them to burst. The oil thus freed is carried off in the current of steam. The steam charged with the essential oil, having passed through the plants to the top of the vat, escapes through a valve to the primary condenser, which consists of a serpentine copper coil over which cold water flows. The distillate after traversing this worm falls into a receiver having an exterior pipe leading from the bottom to a height nearly equal to that of the receptacle. As the distillate flows into the receiver, the water, being heavier than the oil, sinks to the bottom of the vessel and is forced out through the discharge pipe.

In some countries this water, which does not throw off the entire amount of essential oil contained, is returned to the boiler and reconverted into steam and continuously used. This system is employed in the Malaga district only in the preparation of rose oil. The essential oil collects upon the top until the tank is filled. The whole process takes about three hours. As thus distilled from the plants the product obtained is the natural oil, which always contain an insoluble resin. It is in this form that the oil is usually sold.

*Vacuum System of Refining Used.—Prices.*—To give the oil the greatest possible concentration, aroma, and solubility and to free it from foreign substances, it is treated by the vacuum process. The old style was to repeat the process of distillation described above. The newer vacuum system is much superior because the oil boils at a much lower temperature, which prevents the decomposition of the oil. The system employed is identical with the first distillation except that the natural oil instead of the plant is placed in the vat for

distillation and, furthermore, a vacuum is maintained in the vat and coil. The duration of the refining process depends upon the quantity of oil to be distilled. After the aromatic oil has been recovered there remains an oleoresin which is thrown away. This is sometimes found to the extent of over 25 per cent. The refined essential oil has the true plant odor in a high degree, is of the greatest strength, unusual solubility, brilliant and limpid, and is absolutely pure.

*Increased Shipments to America.*—Statistics of the total production of essential oils in this district or of the total exports of such oils from Malaga are not available. However, the following statement, giving in dollars the exports to the United States from 1898 to date, indicates the rapid growth of the industry (the increase of the exports to the United States since the outbreak of the war is worthy of note): In 1898, $179; in 1899, $565; in 1900, none; in 1901, $669; in 1902, $502; in 1903, none; in 1904, none; in 1905, $5,455; in 1906, none; in 1907, $1,231; in 1908, $3,902; in 1909, $3,646; in 1910, $10,445; in 1911, $23,940; in 1912, $72,034; in 1913, $76,966; in 1914, $83,773; in 1915, $137,129; in 1916, $176,-164; and in the first nine months of 1917, $200,329.

The total American imports from all countries of essential oils of the classes shipped from Malaga are shown in the table which follows, the figures representing the American imports for consumption of the oils named during the fiscal years ending June 30, 1915, and 1916:

| Oils. | Year ended June 30 1915. | 1916. |
|---|---|---|
| Fennel | $ 441 | $ 789 |
| Juniper | 7,637 | 1,988 |
| Lavender and spike lavender | 117,039 | 271,815 |
| Origanum | 15,111 | 24,291 |
| Rosemary | 44,636 | 30,504 |
| Thyme | 88,498 | 85,913 |
| Totals of oils named | $273,362 | $415,300 |

By comparing the above with the declared exports of essential oils from Malaga, the great importance of the local supply of these oils will be noted. It is said that the large quantity of rosemary oil imported into the United States is for the denaturing of olive oils at the customhouse, to make them unfit for any but industrial purposes, in which case they are admitted duty free. Large quantities of thyme are now used in America for the extraction of the phenol

therein contained. This latter product has become very scarce since the outbreak of the war, because of the curtailment of the export from certain belligerent countries.

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## WOOD ALCOHOL POISONING.[1]

By A. O. Gettler, Ph.D., and A. V. St. George, M.D.

The prohibition by our government of the manufacture of distilled liquors will unquestionably lead to much "moonshining," adulteration and dilutions of liquors offered to the public. That such is the case, even at this period of the war, is quite evident from the recent poisoning in this city of over thirty persons, six of whom died, with a whisky sold in one of the poorer sections of the city that on analysis proved to contain a considerable amount of wood (methyl) alcohol. Believing that similar cases will occur and with increasing frequency, despite the vigilance of our revenue and state officers, we feel it timely again to warn physicians, coroners and health officers, in order that they may be on their guard in similar cases, as prompt and efficient therapeutic measures must be instituted to hold any hope for recovery of the patient, and furthermore, to detect any violations of the food laws.

It is our belief that the introduction into beverages of the dangerous poison, wood alcohol, is nearly always due to ignorance on the part of the blender. Many physicians and nearly all of the lay public are unacquainted with the chemical and physiologic differences between the relatively innocuous ethyl or grain alcohol and the dangerous methyl or wood alcohol when applied to the body or introduced into it. The refined wood alcohol tastes like ethyl alcohol, and, moreover, is considerably cheaper; hence the adulterator buys the latter, ignorant that severe poisoning, blindness and often death lurk within it.

The pharmacology of this poison has been presented by Pohl and Hunt, and we would refer the reader to their original papers. The violation of the food law and the criminal aspects of our cases are being investigated by the proper authorities, and we shall merely record the findings in the six fatal cases, with points in diagnosis and treatment and methods for detecting the poison.

[1] Reprinted from the *Journal of the American Medical Association.*

Similar but meager histories were obtained, and we were unable to determine the quantities of the poison taken. Four of the patients were admitted to St. Vincent's Hospital, one to Bellevue Hospital, and one patient died in his own home. All had taken whisky a short time previously, and all had complained of violent abdominal pains, incessant vomiting and extreme weakness. Three of the patients complained of blindness. Three of the St. Vincent's patients and the Bellevue patient came into the hospitals in coma; one (Case 1), who was admitted while conscious, said that he had taken whiskey a short time before; soon thereafter he noted intense abdominal pain, vomiting, increasing weakness and dimness of vision; finally he collapsed in the street, and was brought to the hospital, where he died half an hour after admission. The patient had the appearance of being in a severe toxic condition or collapse. The important points in all the physical examinations were slow respirations, dilated pupils, weak and rapid heart action, cold and clammy extremities, and deep coma.

In Case 1, stomach washings were obtained, and also some of the liquor that he had drunk. Both were found to contain wood alcohol.

The examination of the urine during life in Case 2 revealed albumin and casts, and gave a Fehling reduction (aldehyd formation?).

The diagnostic features of the acute type of poisoning are extreme physical weakness, acute gastro-intestinal symptoms, blindness, and deep and prolonged coma, ending in death with cardiac collapse. In the chronic type of cases, blindness is the chief symptom.

A differential diagnosis from epilepsy, and especially all types of coma—uremia, cardiac, diabetic, cerebral, toxic and traumatic—must be made. This frequently is very difficult; but if we bear in mind the important symptoms mentioned above, and obtain a history of whisky drinking, together with an alcoholic breath, we should be suspicious, at least to the extent that we are dealing with some form of alcohol poisoning.

The treatment, to be at all efficacious, must be promptly instituted, and consists essentially of ridding the body of the poison, and supportive measures. The poison is but slowly and incompletely oxidized in the body to more dangerous poisons, namely, formaldehyde and formic acid, and as such very slowly (for four to five days,

and even longer) eliminated by the kidneys. Mayer has demonstrated that the relative toxicity of formic acid is six times greater that that of wood alcohol. For a considerable period of time, the alcohol is excreted unchanged into the stomach and intestine (Bongers), and it is therefore important that early and frequent gastric and rectal lavage be carried out. The stomach washings should be immediately examined for wood alcohol. Intravenous saline or sodium bicarbonate infusions and phlebotomy and transfusions, if the latter can be had quickly, should be administered. In addition, warmth and strong stimulation with strychnine, digitalis, caffeine, camphor, epinephrine, and oxygen must be given. It is said that ethyl alcohol is indicated to replace the methyl, and that it acts as a powerful stimulant; but we doubt this. For the treatment of the chronic form of poisoning, we refer the reader to the article by Buller and Wood, who collected the histories of 275 American cases up to 1904, and to Birch-Hirshfield, who discusses the eye condition in considerable detail.

The postmortems in Cases 1 to 4 were performed by Dr. Otto H. Schultze, and in Cases 5 and 6 by Dr. A. V. St. George, in the presence of Dr. Benjamin Schwartz, coroner's physician. The gross anatomic diagnosis in all the cases were: marked congestion with increased cerebrospinal fluid; marked congestion of all the viscera, and acute pulmonary edema and congestion. In addition, in all cases except Case 2, only dark fluid blood was distributed throughout the heart and vessels. In Case 2 there was an aortic stenosis, and a soft red clot was present in the left heart. The brain in Cases 1 and 5 had a suspicious odor, not, however, distinctive of alcohol; no odor could be detected in the stomach or its contents.

*Results of Tests.*

| | Reduced fuchsin | | Casein test | | Resorcin test | |
|---|---|---|---|---|---|---|
| | Before | After | Before | After | Before | After |
| | Oxidation | | Oxidation | | Oxidation | |
| Case 1, brain | − | + | − | + | − | + |
| Case 1, lavage | − | + | − | + | − | + |
| Case 1, beverage | − | + | − | + | − | + |
| Case 2, brain | − | + | − | + | − | + |
| Case 3, brain | − | + | − | + | − | + |
| Case 4, brain | − | + | − | + | − | + |
| Case 5, brain | − | + | − | + | − | + |
| Case 5, urine 220 Cc. | − | + | − | + | − | + |
| Case 5, blood 490 Cc. | − | + | − | + | − | + |

In case 6 there was, in addition, a chronic pulmonary tuberculosis. In Case 5 there was marked parenchymatous degeneration of the kidneys and liver. Microscopic section of heart, kidney and liver did not show an increased amount of fat. Pohl reported that he found an increase in the amount of fat in the liver. Sections of the optic, vagus and phrenic nerves show marked degeneration of these structures. Parts of the brain in all of the six cases were examined chemically according to the method given below. We selected the brain tissue for the performance of our tests, because of the assumption that the poison has a selective action on nerve tissue, as is indirectly proved by the blindness and deep coma.

*Chemical Analysis of Brain.*—The sample was finely ground, and water was added to make a thin suspension; it was placed in a distillation flask; 3 drops of liquid petrolatum were added to prevent frothing; it was acidified with tartaric acid and distilled with steam. Thirty Cc. of the distillate were collected. Of this, 5 Cc. were placed in a test tube and oxidized by the plunging of a red hot copper spiral into it six or eight times. This oxidation is for the purpose of converting the methyl alcohol to formaldehyde.

The oxidized material as well as a little of the original distillate was tested by the following three methods:

*Reduced Fuchsin Test.*—To 2 Cc. of each of the distillates (both oxidized and nonoxidized), 3 drops of (colorless) reduced fuchsin were added. A red color develops if an aldehyd is present. This test is not specific for formaldehyde, but is given by the aldehyd group in general.

*Casein Test.*—To 2 Cc. of each of the distillates (both oxidized and nonozidized), an equal amount of milk, then an equal volume of hydrochloric acid, and 2 drops of ferric chloride are added. This is brought to a boil. If formaldehyde is present, a violet to red color develops. This test is specific for formaldehyde and is very delicate, detecting formaldehyde in dilutions of 1:250,000.

*Resorcin Test.*—To 2 Cc. of each of the distillates (both oxidized and nonoxidized), a few drops of 0.5 per cent. resorcin are added, and stratified on sulphuric acid. In the presence of formaldehyde a red ring develops. This test is specific for formaldehyde.

The brain in every one of the six cases was found to have methyl alcohol present. The tests before oxidation were negative, showing that no oxidation to aldehyd occurred in the brain.

In Case 5, blood and urine also were available. These were ex-

amined according to the method given; both the blood (490 Cc. of blood being used) and the urine (220 Cc.) showed methyl alcohol but no formaldehyde.

It is important to note that the refined wood alcohol, in addition to being used as a diluent of whisky, is frequently used in the preparation of essence of peppermint, Jamaica ginger, lemon extract, punch, cologne, liniments, Florida water, bay rum, witch hazel and in the arts in lacquers, varnishes, etc.

### Conclusions.

Physicians and health officers should warn the public of the dangers existing in the preparations of the kind enumerated above when applied to or introduced directly or by inhalation into the body. European authorities have shown that the denatured ethyl alcohol serves every purpose in medicine and in the arts as well as wood alcohol or better. We suggest, therefore, that legislation be enacted bringing about the prohibition of the sale of wood alcohol for domestic purposes, similar to laws now obtaining in England and Germany.

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## BARIUM SULPHATE FOR X-RAY WORK.[1]

The use of pure barium sulphate for X-ray work has already been referred to (see *Prescriber*, 1917, Feb., p. 52). It is inexpensive, being much cheaper than bismuth, it is opaque to the rays, and if perfectly pure there is no danger of toxicity. The drawback is its density and consequent tendency to fall to the bottom when suspended in any liquid or semi-liquid medium.

A formula for a barium meal is given in the B. P. Codex as follows:

| | |
|---|---|
| Barium sulphate | 150 Gm. |
| Corn flour | 15 Gm. |
| Sugar | 15 Gm. |
| Cocoa | 20 Gm. |
| Water | 500 Mils. |

These ingredients are to be boiled together, and if the mixture is properly made the barium salt is well diffused, and there is little or no risk of it forming a deposit in the stomach or bowel.

[1] From *The Prescriber.*

R. Bensaude and M. Terrey (*Presse medicale,* 1917, 25, 168; Mar. 22) recommend a gelatin cream containing 50 per cent. of barium sulphate; this can, they say, be kept without any tendency to precipitation for a long time. The preparation of the sulphate requires great care, beginning with pure recrystallized barium chloride, which is precipitated completely with excess of sodium sulphate; the precipitate is then washed many times to eliminate every trace of chloride. Incorporated with a gelatin medium it forms a velvety cream in which the barium salt is so finely suspended that it deposits very slowly indeed. An emulsion containing 200 Gm. of this cream, 40 Gm. of syrup of acacia, and distilled water to 250 Mils., may me made without trouble, and is stable for several hours. A more stable emulsion may be made from 200 Gm. of the barium-gelatin cream, with tragacanth, 5 Gm.; acacia, 2 Gm.; glycerin, 40 Gm.; and water to one liter. This mixture can be sterilized and when a deposit forms upon keeping, it is at once diffused by agitation. A barium meal may be prepared by mixing barium-gelatin cream, 200 Gm.; with powdered soluble chocolate, 50 Gm.; powdered agar, 10 Gm.; and boiling water, 150 Mils. Very good results are reported from this preparation.

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## THE ESTIMATION OF "WOODY-FIBER" IN FEEDING-STUFFS.[1]

By J. A. Voelcker and E. W. Voelcker.

It is very desirable that in an "empirical" method of analysis such as is involved in the estimation of woody-fiber there should be agreement as to the strengths of acid and alkali used in the process. In this country the leading agricultural analysts, following the method adopted by the late Dr. Augustus Voelcker, F.R.S., use 2 per cent. solutions of sulphuric acid and caustic alkali for the hydrolysis. The American and Continental official methods, however, prescribe solutions of the strength of 1.25 per cent. only, and, owing to the unfortunate tendency in this country to favor the adoption of foreign methods of analysis, these find their way into textbooks and are liable to be regarded as "official" here. In the case of such feeding-stuffs as the cereal grains and their offals,

[1] From *The Analyst.*

variations within limits of the strength of acid and alkali used do not materially affect the results obtained; but with oil cakes, especially those made from seeds, with a more or less hard "husk," very wide differences in results may arise, according as to whether 1.25 or 2 per cent. acid and alkali have been employed. In the case, for example, of palm nut and similar cakes it is impossible to properly effect the hydrolysis with the weaker solutions, and even with the 2 per cent. solutions vigorous boiling for half an hour is essential. It is best to operate on the material after extraction of the oil with ether. Three grammes of the ground sample are extracted with ether and then boiled vigorously for half an hour with 125 Cc. of 2 per cent. sulphuric acid, the volume of the liquid being kept constant during the operation. The liquid is diluted to 400 or 500 Cc. with water, and allowed to settle. The liquid is then poured through a filter—preferably a piece of fine linen—the residue is washed on to this, and then transferred to the original boiling vessel, and boiled for half an hour with 125 Cc. of 2 per cent. caustic alkali solution. The liquid is diluted as before, allowed to settle, and the insoluble fiber collected, either on a counterpoised filter or on the linen filter, from which it can readily be completely removed. The fiber is washed to free it from alkali, then treated with a little dilute acid, followed by water, and finally with alcohol. The fiber is dried at 100° C. to constant weight, and burnt, any mineral matter found being deducted from the weight of the fiber.

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## SELECTED PHARMACEUTICAL FORMULAS FROM THE CHEMIST'S CALENDAR.

(*Continued from Page 207.*)

### Foot-Liquid.

| | |
|---|---|
| Liquid formaldehydi | ℥ixss |
| Sp. rosmarini | ℥ss |

A teaspoonful to a basin of water.

Very good for hardening the feet and healing blisters. Much appreciated in Army circles.

### Acid Aperient Mixture.

| | |
|---|---|
| Magnesii sulphat. | ℥iij |
| Acid. sulph. dil. | ℥iij |
| Syrup. aurantii | ℥iss |
| Aq. menth. pip. ad | Oj |

Dose: A dessertspoonful or a tablespoonful with water twice or thrice daily.

FOR PERSPIRING HANDS.

| | |
|---|---|
| Lotion acid carbolic. (1 in 40) | ℥v |
| Liq. formaldehyd. | ʒss |
| Acid. acetic. | ʒj |
| Ess. Parma violet | ʒj |
| Aq. dest. ad | ℥vj |

To be well rubbed into the palms of the hands and between the fingers several times daily.

DRY SHAMPOO FOR HAIR.

| | |
|---|---|
| Orris powder | ʒij |
| Rice-flour | ʒi, ʒvj |
| Coumarin | Gr. ij |

Mix intimately.

To be sprinkled among the hair and brushed off with a clean brush, prolonging the draw of the brush to the end of the hairs.

This resembles the best type of dry shampoo, and proves satisfactory. More complex formulas do not appear to afford better results.

LIQUID DRY SHAMPOO.

| | |
|---|---|
| Saponis dur. | ʒj |
| Spirit. meth. (industrial) | ℥viij |
| Aq. destill. | ℥iij |
| Macerate for three or four days, filter and add: | |
| Liq. ammoniæ | ʒj |
| Sp. rosmar. | ʒss |

To be sprinkled over the hair, brushed through, and then rubbed off with a dry towel.

Reported to be very refreshing after a hot day, and to leave the hair "just nice."

BATH-LIQUID.

| | |
|---|---|
| Liquid ammoniæ | ʒj |
| Liq. extract. quillaiæ | ʒiss |
| Synthetic bergamot | ʒss |
| Synthetic musk | Gr. ijj |
| Aq. ad | ʒiv |

To be added to a bath. Softens the water, promotes a fine lather, and scents the skin. In favor among ladies. The perfume can be varied to suit individual preference.

### Bath or Toilet Ammonia.

| | |
|---|---|
| Saponis communis | Gr. viij |
| Liq. ammon. fort. | ʒvj |
| Sp. lavand. | ʒij |
| Aq. dest. ad | ʒxx |

About a wineglassful to a bath; about a dessertspoonful to a toilet-basin.

Renders washing pleasant when the water is hard; with soft water it is stimulating. The scent can be varied or increased; an equivalent quantity of ess. of sweet-pea or ess. of Parma violet makes an agreeable change.

### Sunburn-Lotion.

| | |
|---|---|
| Blanched almonds | ʒj |
| Borax | Gr. xx |
| Simp. tincture of benzoin | ♏L |
| Orange-flower water | ʒiiiss |
| Sol. of hydrogen peroxide | ʒss |

Bruise the almonds and triturate with successive portions of the orange-flower water in which the borax has previously been dissolved; strain through muslin, and add the tincture of benzoin and the solution of hydrogen peroxide.

Good as a protective before going out or as a cooling application on return.

### Sunburn Cream.

| | |
|---|---|
| Adipis lanæ hydros. | ʒj |
| Paraffin. liq. | ʒij |
| Liq. hydrog. perox. | ʒiij |
| Synthetic otto | Gtt. v |

Mix the lanoline and liquid paraffin together in a warm mortar, incorporate the solution of hydrogen peroxide, and finally add the otto.

Makes a very nice toilet lanoline to send out in pots. Should be used sparingly and well rubbed in.

### Poison for Rats and Mice.

| | |
|---|---|
| Barium carbonate | ℥iv |
| Biscuit-flour | ℥iij |
| Oatmeal | ℥j |
| Oil of anise | ♏v |
| Synthetic musk | Gr. j |

Beef dripping sufficient to make a paste.

Very effective, and does not contain a scheduled poison. Pieces the size of a bean can be laid in holes and runs.

A few pieces of naphthaline placed in mouse-holes in a house have the effect of causing the little rodents to abandon their quarters.

### Worm-Ball for Dog.

| | |
|---|---|
| Pulv. arecæ | Gr. xxx |
| Pulv. santonin. | Gr. ij |
| Pulv. tragacanth. | Gr. j |

Syrup. glucosi q. s. to form a bolus of pill consistence.

This is a suitable dose for a dog of 15 or 20 lb. in weight.

### Quinine Hair-Tonic.

| | |
|---|---|
| Quin. hydrochlor. | ℥j |
| Cudbear | Gr. xx |
| Aq. chlorof. | ℥xx |
| Aq. rosæ | ℥xl |
| Glycerin | ℥j |

Macerate for four days, shaking twice daily, and filter.

To be sprinkled on the scalp and rubbed in every morning.

### Shampoo Powder.

| | |
|---|---|
| Pulv. saponis | ℥x |
| Pulv. sodii carb. com. exsiccat. | ℥ij |
| Pulv. boracis | ℥ij |
| Synthetic otto | ℥ss |
| Synthetic musk | Gr. v |

About a third of an ounce is sufficient to put up for one shampoo. Should for use be dissolved in a pint or so of warm water, and this solution used to soak the hair and get up a froth.

### Casein Nerve-Food.

| | |
|---|---|
| Sodium glycerophosph. | Gr. ij |
| Casein | ℥iss, Gr. viij |

To be added to a bath. Softens the water, promotes a fine lather, and scents the skin. In favor among ladies. The perfume can be varied to suit individual preference.

### Bath or Toilet Ammonia.

| | |
|---|---|
| Saponis communis | Gr. viij |
| Liq. ammon. fort. | ʒvj |
| Sp. lavand. | ʒij |
| Aq. dest. ad | ʒxx |

About a wineglassful to a bath; about a dessertspoonful to a toilet-basin.

Renders washing pleasant when the water is hard; with soft water it is stimulating. The scent can be varied or increased; an equivalent quantity of ess. of sweet-pea or ess. of Parma violet makes an agreeable change.

### Sunburn-Lotion.

| | |
|---|---|
| Blanched almonds | ʒj |
| Borax | Gr. xx |
| Simp. tincture of benzoin | ♏L |
| Orange-flower water | ʒiiiss |
| Sol. of hydrogen peroxide | ʒss |

Bruise the almonds and triturate with successive portions of the orange-flower water in which the borax has previously been dissolved; strain through muslin, and add the tincture of benzoin and the solution of hydrogen peroxide.

Good as a protective before going out or as a cooling application on return.

### Sunburn Cream.

| | |
|---|---|
| Adipis lanæ hydros. | ʒj |
| Paraffin. liq. | ʒij |
| Liq. hydrog. perox. | ʒiij |
| Synthetic otto | Gtt. v |

Mix the lanoline and liquid paraffin together in a warm mortar, incorporate the solution of hydrogen peroxide, and finally add the otto.

Makes a very nice toilet lanoline to send out in pots. Should be used sparingly and well rubbed in.

### Poison for Rats and Mice.

| | |
|---|---|
| Barium carbonate | ℥iv |
| Biscuit-flour | ℥iij |
| Oatmeal | ℥j |
| Oil of anise | ♏v |
| Synthetic musk | Gr. j |

Beef dripping sufficient to make a paste.

Very effective, and does not contain a scheduled poison. Pieces the size of a bean can be laid in holes and runs.

A few pieces of naphthaline placed in mouse-holes in a house have the effect of causing the little rodents to abandon their quarters.

### Worm-Ball for Dog.

| | |
|---|---|
| Pulv. arecæ | Gr. xxx |
| Pulv. santonin. | Gr. ij |
| Pulv. tragacanth. | Gr. j |

Syrup. glucosi q. s. to form a bolus of pill consistence.

This is a suitable dose for a dog of 15 or 20 lb. in weight.

### Quinine Hair-Tonic.

| | |
|---|---|
| Quin. hydrochlor. | ℥j |
| Cudbear | Gr. xx |
| Aq. chlorof. | ℥xx |
| Aq. rosæ | ℥xl |
| Glycerin | ℥j |

Macerate for four days, shaking twice daily, and filter.

To be sprinkled on the scalp and rubbed in every morning.

### Shampoo Powder.

| | |
|---|---|
| Pulv. saponis | ℥x |
| Pulv. sodii carb. com. exsiccat. | ℥ij |
| Pulv. boracis | ℥ij |
| Synthetic otto | ℥ss |
| Synthetic musk | Gr. v |

About a third of an ounce is sufficient to put up for one shampoo. Should for use be dissolved in a pint or so of warm water, and this solution used to soak the hair and get up a froth.

### Casein Nerve-Food.

| | |
|---|---|
| Sodium glycerophosph. | Gr. ij |
| Casein | ℥iss, Gr. viij |

Powder the sodium glycerophosphate and mix lightly with the casein.

Dose: One or two teaspoonfuls at mealtimes.

### Chilblain-Paint.

| | |
|---|---|
| Menthol | ʒij |
| Ol. ricini | Gr. xv |
| Acid. tannic. | ʒj |
| Sp. rect. | ʒij |

To be painted over the chilblains morning and evening, and more frequently when itching.

### For Broken Chilblains.

| | |
|---|---|
| Phenol | Gr. xl |
| Ung. acid. boric., B.P. 1898 | ʒiij, ʒiij, Gr. xl |
| Zinci oxid. | ʒss |

Make a smooth ointment.

To be applied on lint or linen. The dressing to be changed night and morning. Once a day dribble hot carbolic lotion, 1 in 60, from a height onto the sore before applying a new dressing.

### Asthma Powder.

| | |
|---|---|
| Pulv. fol. stramonii, | |
| Pulv. potass. nitratis, | |
| Pulv. fruct. anisi | partes æquales |

Dry each separately and mix intimately. About as much as will lift on a shilling made into a little cone and ignited gives off a smoke, which is to be inhaled when an attack is imminent or has come on.

### Inhalation for Cold in the Head.

| | |
|---|---|
| Menthol | ʒss |
| Ol. pini | ♏xx |
| Acid. benzoic. | Gr. xx |
| Pulv. benzoin. | ʒij |
| Kieselguhr | ʒss |

Mix intimately.

Half a teaspoonful of this in a jug with half a pint of hot water makes a very pleasant and useful inhalation.

### Toothache-Essence.

| | |
|---|---|
| Menthol | ℥j, Gr. xx |
| Tinct. pyrethri | ℥j |
| Tinct. hyoscy. | ℥j |
| Tinct. bellad. | ℥ss |
| Sp. rect. | ℥iij |

To be rubbed on the gum over the ache, or placed in any cavity there may be.

### Lip-Salve.

| | |
|---|---|
| White beeswax | 2 Oz. |
| Spermaceti | 1 Oz. |
| Sesame oil | 4 Oz. |
| Honey | 2 Oz. |
| Oil of bergamot | 120 Min. |

Melt the wax and spermaceti, add the honey, and heat together; then gradually pour in the oils, and stir until cold. May be colored with alkanet or vermilion.

### Oxygen Skin-Lotion.

| | |
|---|---|
| Zinc oxide | 120 Gr. |
| Powdered tragacanth. | 30 Gr. |
| Glycerin or honey | 1 Oz. |
| Ess. white rose | 120 Min. |
| Solution of hydrogen peroxide | 2 Oz. |
| Distilled water to | 12 Oz. |

Stir the tragacanth into the essence, add quickly 4 Oz. of the water, and then the remainder. Triturate well the zinc oxide with the glycerin, incorporate gradually the tragacanth mucilage, and finally the solution of hydrogen peroxide.

This is smeared over the neck and shoulders at night and allowed to remain on over night. Used for several consecutive nights it improves the appearance of the skin for evening dress.

# DISINFECTION WITH FORMALDEHYDE. A SUBSTITUTE FOR THE PERMANGANATE–FORMALIN METHOD.[1]

By C. G. Storm.

The method proposed by H. D. Evans and J. P. Russell in 1904[2] for the rapid liberation of formaldehyde gas from its water solution, the "formalin" of commerce, in a condition suitable for practical disinfection, has been found by numerous investigators to be superior to most of the other known methods of formaldehyde disinfection, as regards simplicity, rapidity, cost and efficiency. This method consists in pouring the formalin quickly upon crystals of potassium permanganate contained in any suitable metallic vessel (for example, a water bucket), the oxidation of a part of the formaldehyde furnishing sufficient heat to cause rapid evaporation of the remainder of the formaldehyde together with the water.

The permanganate method has found very general application and is widely used in this country in practical disinfection work. Its use has, however, received a serious set-back by reason of the present scarcity of potassium permanganate and the resulting excessive cost of this chemical. Prior to the war in Europe, potassium permanganate was obtainable in this country at prices ranging usually from 9 to 10 cents per lb. It is now obtainable only at many times its former price, having held at approximately $4.00 to $4.25 per lb. for the past six months.

This fact has impressed the writer with the desirability of publishing a note regarding an analogous method for generating formaldehyde, devised by him in October, 1911, which it is believed has never been proposed for practical use, which preliminary tests indicate to be safer, as rapid, and almost as simple of operation, and which will be much less expensive, owing to the relatively low price of the material used.

The new method depends upon the action between the water

[1] Reprinted from *The Journal of Industrial and Engineering Chemistry*, February, 1918.

[2] H. D. Evans and J. P. Russell, "Formaldehyde Disinfection," *13th Ann. Rept.*, State Board of Health of Maine, and *J. Am. Chem. Soc.*, 27 (1905), 714. See also Daniel Base, "Formaldehyde Disinfection," *J. Am. Chem. Soc.*, 28 (1906), 964–96.

solution of formaldehyde and a soluble chlorate, and is apparently analogous to the permanganate method, in that the oxidation of a part of the formaldehyde furnishes a sudden evolution of heat which serves to vaporize the remainder of the formaldehyde. It was suggested to the writer in the course of analysis of a potassium chlorate explosive. On adding formalin to the water solution of the explosive and heating the mixture, a violent evolution of gas resulted, increasing in intensity even after the tube containing the mixture was removed from the flame of the burner. The gas evolved was largely formaldehyde, apparently liberated from its solution by the heat generated in the oxidation of a part of the formaldehyde by the chlorate.

An examination of the solution remaining after the reaction had subsided showed the presence of large amounts of chloride which had resulted from reduction of the chlorate. Repeated trials showed that the violent evolution of gas resulted only from concentrated solutions, but that the reduction of the chlorate to chloride, with a corresponding oxidation of formaldehyde, took place even in the case of very dilute solutions of chlorate. It has been demonstrated that under proper conditions this reaction is quantitative, and the results of the study of this quantitative method for determining chlorates will shortly be published.

The object of this paper is merely to call attention to what it is hoped will be a satisfactory substitute for the permanganate method of disinfection, and to offer an opportunity for a more complete study of the method, the writer's investigation having necessarily been quite incomplete because of lack of time and facilities for conducting work of this nature.

Potassium permanganate reacts immediately on coming in contact with formalin at ordinary temperatures, and if the permanganate is finely powdered instead of crystalline, the reaction may be violently explosive in character.[3] If formalin is poured on crystals of sodium or potassium chlorate, no action results until the mixture is warmed by application of external heat to about 65° C. This may be considered as a disadvantage, but as a matter of fact the reaction may be started with very little difficulty. The chlorate and formalin are placed together in a suitable metal container, such as a water bucket, of sufficient size to prevent the reaction mixture from foaming over, and the bucket, properly weighted so it will not

[3] G. B. Frankforter and R. M. West, *J. Am. Chem. Soc.*, 28 (1906), 1234.

float, placed in a large shallow pan (an ordinary dish pan will answer the purpose) containing water heated to about the boiling point.

The mixture becomes heated to the reaction temperature in a few minutes, when bubbles of gas begin to be evolved, this evolution increasing rapidly until it is so violent that the mixture may foam over the top of the bucket. The action is completed in 2 or 3 min., and with the proper proportion of chlorate and formalin the residue remaining in the bucket is practically dry and consists chiefly of chloride together with some unreduced chlorate.

Sodium chlorate seems to give just as satisfactory results as potassium chlorate, and has the distinct advantage of costing less than one half as much as the latter. Potassium chlorate is now quoted at 50 to 55 cents per lb., while sodium chlorate is listed at 24 to 25 cents.

Several investigators have attempted to determine what proportion of formaldehyde used in the permanganate process is liberated as gas and what proportion is oxidized by the reaction. Frankforter and West[4] obtained an evolution of 62 per cent. to 75 per cent. of the formaldehyde from formalin by this process in a long series of experiments under laboratory conditions, using glass apparatus and absorbing the evolved gas in water, the strength of the resulting solution being determined. D. Base,[5] in experiments with the process on a practical scale, used a specially prepared room of 2,000 cu. ft. capacity, determining the amount of formaldehyde gas in the room by drawing 5- to 10-liter samples through standard KCN solution, adding excess of standard $AgNO_3$ solution and titrating the excess of the latter with sulphocyanate. Base found that not over 40 per cent. of the total amount of formaldehyde used as formalin was evolved in the state of gas in the room.

It has been suggested that the reaction between formaldehyde and potassium permanganate is probably as follows:

$$4KMnO_4 + 3HCHO + H_2O = 4Mn(OH)_2 + 2K_2CO_3 + CO_2.$$

It is, however, likely that other reactions proceed at the same time, in which part of the formaldehyde is oxidized to formic acid. Assuming this reaction, however, it is calculated that with the proportions recommended by Evans and Russell (100 Cc. of 40 per cent. formalin to 37.5 Gm. $KMnO_4$) 5.34 Gm. of formaldehyde, or about

[4] *J. Am. Chem. Soc.*, 28 (1906), 1234.
[5] *Ibid.*, 28 (1906), 964.

13.35 per cent. of the formalin, would be oxidized by the $KMnO_4$. With the proportions recommended by Base (100 Cc. of formalin to 50 Gm. $KMnO_4$) 7.12 Gm. HCHO or 17.8 per cent. of the formalin would be destroyed. These figures are considerably lower than those found in the investigations mentioned above. In any event it is apparent that in the permanganate process a considerable part of the formaldehyde used as formalin is destroyed by oxidation, the reaction supplying the heat which causes the rapid volatilization of the remainder.

It may be assumed that the behavior of the chlorate with the formalin is entirely analogous to that of the permanganate, and that the reaction occurs according to the equation

$$2KClO_3 + 3HCHO = 2KCl + 3H_2O + 3CO_2.$$

Experiments have shown that the proportion of 25 Gm. of chlorate to 100 Cc. of formalin is approximately the one giving best results, that is, with these proportions there is practically no liquid left in the residue after the reaction subsides, the formaldehyde being either driven off as gas or oxidized and the water evaporated at the same time.

A simple calculation shows that, according to the above reaction, 25 Gm. $KClO_3$ will theoretically oxidize 9.18 Gm. HCHO or nearly 23 per cent. of the formaldehyde in the 100 Cc. of formalin, leaving the remaining 17 per cent. to be volatilized. It is probable, however, that other reactions occur, such as

$$KClO_3 + 3HCHO = KCl + 3HCOOH.$$

In fact, appreciable amounts of formic acid, as well as $CO_2$, are evolved by the reaction of formalin with either permanganate or chlorate.

In an attempt to determine in a simple manner the best proportions of formalin and chlorate, a series of roughly quantitative experiments were made, using varying proportions of the two materials. A weighed amount of powdered $KClO_3$ was treated in a beaker with a weighed amount of the 40 per cent. formalin and the beaker immersed in hot water in order to start the reaction. The residue left in the beaker after the reaction had ceased was dissolved in water and titrated with standard solution of silver nitrate to determine the amount of chloride present. From the amount of KCl found, the weight of formalin representing the formaldehyde

destroyed in the reduction of $KClO_3$ to KCl was then calculated from the reaction

$$2KClO_3 + 3HCHO = 2KCl + 3CO_2 + 3H_2O.$$

The results of these tests are shown in the following table. It is to be noted that if the reaction

$$KClO_3 + 3HCHO = KCl + 3HCOOH$$

is assumed, the calculated amounts of formaldehyde consumed will be just twice those given in the table.

*Tests of Residue Remaining after Reaction between Formalin and Potassium Chlorate.*

| Test No. | $KClO_3$ Used, Grams. | Formalin Used, Grams. | Calc. $KClO_3$ Reduced, Grams. | HCHO Oxidized (Equiv to KCl Found), Grams. | Formalin (40%) Equiv. to HCHO Oxidized, Grams. |
|---|---|---|---|---|---|
| 1 | 7 | 12 | 3.917 | 1.439 | 3.59 |
| 2 | 6 | 12 | 3.817 | 1.402 | 3.50 |
| 3 | 6 | 12 | 3.620 | 1.329 | 3.33 |
| 4 | 5 | 12 | 3.750 | 1.375 | 3.44 |
| 5 | 4 | 12 | 3.726 | 1.369 | 3.42 |
| 6 | 3 | 12 | 2.990 | 1.098 | 2.75 |
| 7 | 2 | 12 | 2.010 | 0.738 | 1.85 |

In Tests 5 and 6 a very small amount of liquid remained in the residue after the reaction; in Test 7 an appreciable amount of liquid remained and a determination of formaldehyde showed 1.104 Gm. HCHO, equal to 2.76 G. of 40 per cent. formalin. In Tests 1 to 5, inclusive, where the weight of $KClO_3$ was at least one third of the weight of the formalin, the amount of HCHO oxidized was fairly constant, the KCl found indicating that only part of the $KClO_3$ had been reduced. In Tests 6 and 7 the excess of formalin was such that practically complete reduction of the chlorate was effected and the amount of formalin oxidized much less.

A number of qualitative tests were made using formalin and sodium chlorate in proportions varying from 6:1 to 2:1, the maximum temperatures reached during the reaction being noted. With the ratios 2:1, 2.5:1, and 3:1, this temperature was 108–109° C., while with lower proportions of chlorate (4:1 and 6:1) the temperature was slightly less, 104° to 105° C. In each case the reaction started at 60–65° C., was violent at about 75° C., and lasted only about 30 seconds, the maximum temperature being indicated near the end of the reaction.

The writer hopes that comparisons of the actual disinfecting efficiencies of the permanganate and chlorate methods will be made by those who may be interested in the practical side of the question and that the chlorate method may be found to be of some use.

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## CHLOROCOSANE.[1]

Chlorocosane is a solvent for dichloramine-T, which is proposed for the purpose by Drs. H. D. Dakin and E. K. Dunham. The method of preparation is as follows: Paraffin wax, preferably melting at 50° C. or higher, is placed in round-bottomed flasks and heated to about 120°. Two flasks connected in series, each containing a half kilo of the wax, may be conveniently used. A rapid current of chlorine from a cylinder of the liquefied gas is then passed through the molten wax in the flasks, each of which is provided with a thermometer and the necessary glass tubes. The temperature should be controlled within the limits of 125°–140°. The first flask in which the reaction is most vigorous will require but little heating. Chlorination is continued until the contents of the flasks have increased in weight 45 to 55 per cent. of the weight of wax taken. It will be found convenient to complete the chlorination of the first flask and then remove it, and transfer the second to its place, putting a fresh flask of paraffin wax after it. In order to avoid undue discoloration of the product, a minimum amount of rubber tubing should be used for the connections. Wide glass tubing should be used for passing the gases, and the ends of the delivery tubes are preferably blown into bulbs provided with a number of fine orifices to promote good contact between the gas and oil. Hydrochloric acid is, of course, evolved freely during the reaction. After the requisite amount of chlorine has been absorbed, the oil, while still warm, is shaken vigorously with 5 per cent. of its weight of dry sodium carbonate, and then filtered through a dry fluted paper. The clear oil, which has a light yellow or sherry color and is slightly heavier than water, is then ready for use. It possesses a viscosity intermediate between that of olive oil and castor oil, has almost no odor, and is perfectly bland when sprayed up the nose or placed on skin or wounds. Chlorocosane at room temperature can dissolve from 8 to 10 per cent. of dichloramine-T. For wound-

[1] British Medical Journal, reprinted from *The Chemist and Druggist.*

treatment a 7.5 to 8 per cent. solution is strong enough for all purposes, and is most conveniently prepared as follows: A portion of the oil—for example, a quarter—is heated to 75° or 80° C. and the weighed amount of solid dichloramine-T is then added. The latter substance will promptly melt and dissolve in the warm oil, which is then at once diluted with the remainder of the oil and the whole well mixed. If necessary, the solution can be filtered, but with pure materials this is not required. The clear solution should be at once stored in small amber, corked bottles, and not exposed to unnecessary heat.

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## CURRENT LITERATURE.

### SCIENTIFIC AND TECHNICAL ABSTRACTS.

DETERMINATION OF CREATININ IN THE BLOOD.—Tchertkoff describes a technic with which it is possible to detect grave insufficiency of the kidney by the creatinin content of the blood. With only 15 Cc. of blood serum it is possible to ascertain the creatinin content and also the indican and urea content, and thus obtain deep insight into the kidney functioning. He says that the normal creatinin content does not surpass 18 Mg. to the liter of serum. Above 25 Mg., retention is certain. First there is retention of urea, then of creatinin, and later of indican. As the kidney functioning improves they disappear in reverse order. The serum is freed from albumins with an equal quantity of a 20 per cent. solution of trichloracetic acid. After agitation it is filtered, and to 2.5 Cc. of the filtrate is added an equal amount of a saturated solution of picric acid and 1 Cc. of a 20 per cent. solution of sodium hydroxid. By the end of fifteen minutes the fluid changes in tint to a yellow or orange, proportional to the creatinin content. If the tint in ten minutes is still light yellow, the creatinin content is probably within normal range. Above 35 Mg. to the liter, the tint shows orange or even red. With a colorimeter the exact figure can be determined. (*The Journal of the American Medical Association.*)

VOLUMETRIC ESTIMATION OF MERCURIC OXYCYANIDE.—Mercuric oxycyanide, used as a disinfectant, particularly for the hands, may be analyzed as follows: A weighed quantity of 0.3 to 0.4 Grm.

of the salt is dissolved in 50 Cc. of cold water, and the liquid, after treatment with 1 Grm. of sodium chloride and a drop of 0.2 per cent. methyl-orange solution, is titrated with N/10 hydrochloric acid until it turns red. After the volume of acid required is read, 2 Grms. of potassium iodide are added to the solution, and the yellow liquid thus obtained is again titrated with N/10 hydrochloric acid as before. From the two volumes of acid required the percentages of oxycyanide and cyanide in the salt may be calculated, the reactions proceeding according to the equations: (1) $HgO, Hg(CN)_2 + 2HCl = HgCl_2 + Hg(CN)_2 + H_2O$, and (2) $Hg(CN)_2 + 4KI + 2HCl = HgK_2I_4 + 2KCl + 2HCN$.—A. Tagliavini. Boll, *Chim. Farm.*, 1917, 56, 297–299; through *J. Chem. Soc.*, 1917, 112, II, 510.) From *Food and Drug Analysis* through *The Analyst.*

Estimation of Mercury in Galenical Preparations.—The method of destroying organic matter previously employed in analyzing protein preparations containing silver is applied to the determination of mercury in galenical preparations. The substance (0.3 to 0.5 Grm.) is heated with concentrated sulphuric acid (10 Cc.) and nitric acid (sp. gr. 1.4, 3 Cc.) until reddish-yellow vapors are not further evolved, the liquid becomes clear and colorless, and the flask is filled with sulphuric acid fumes. If the vapors still smell of sulphur dioxide, treatment with nitric acid (3 Cc.) is repeated. After being cooled, water (25 Cc.) is added, which is removed by evaporation. The cold solution is treated with water (15 Cc.) and potassium permanganate solution until a permanent pink coloration is produced; the latter is discharged with ferrous sulphate, the solution diluted with water (75 Cc.) and titrated with N/10 ammonium thiocyanate solution in the presence of ferric alum. The method is suitable for organic and inorganic preparations of the oxides of mercury, but not for such as contain the haloids. It can also be used for estimating mercury in plasters and pills, and in ointments, provided that they are tolerably free from paraffin.—H. Wastenson. (*Pharm. Post.*, 1917, 50, 125–126; through *J. Chem. Soc.*, 1917, 112, II, 509.) From *Food and Drug Analysis*, through *The Analyst.*

Volumetric Estimation of Zinc Phenolsulphonate.—In the presence of bromine and an acid, zinc phenolsulphonate reacts in accordance with the following equations: $(OH \cdot C_6H_4 \cdot SO_3)_2Zn$,

$7H_2O + H_2SO_4 = ZnSO_4$, $7H_2O + 2OH \cdot C_6H_4 \cdot SO_3H$ and $2OH \cdot C_6H_4SO_3H + 12Br = 2C_6H_2Br_3 \cdot OH + 6HBr$. Exactly 0.5 Grm. of the crystallized zinc phenolsulphonate is dissolved in water and the solution made up to 500 Cc. In a flask holding about 300 Cc. and fitted with a ground stopper, 50 Cc. of this solution are mixed with 50 Cc. of 0.6 per cent. potassium bromide solution and 50 Cc. of 0.1671 per cent. potassium bromate solution, 5 Cc. of concentrated sulphuric acid being then added, and the flask again shaken and left closed in a dark place at about 25° C. for three hours. Ten Cc. of 10 per cent. potassium iodide solution, recently prepared, are next added and, after the lapse of an hour, the iodine liberated is determined by titration with N/10 sodium thiosulphate. The number of Cc. of the thiosulphate used is subtracted from that required in a blank test with the 50 Cc. of bromide and 5 Cc. of bromate alone: the remainder, multiplied by 0.00447, gives the weight in Grms. of crystallized zinc phenolsulphonate in the 50 Cc. of solution taken.—G. Adanti. (*Boll. Chim. Farm.*, 1917, 56, 317–318; through *J. Chem. Soc.*, 1917, 112, II, 517; through *The Analyst.*)

Detection of Foreign Oils in Castor Oil used for Lubricating Aviation Engines.—For the detection of foreign oils in castor oil the only method sufficiently sensitive is the determination of the acetyl value, which will indicate the presence of 1 to 2 per cent. of arachis or other oils. The solidification-point and the solubility in alcohol will only indicate the presence of 5 per cent. of arachis oil, whilst Bellier's arachidic acid test will not detect less than 7.5 per cent. of arachis oil. A more sensitive test is to determine the turbidity temperature of a solution of 1 volume of the oil in 5 volumes of 95 per cent. alcohol. In the case of pure castor oil the solution can be cooled below —20° C. without becoming turbid, whereas in the presence of arachis oil the solution shows a turbidity at temperatures varying with the proportion of foreign oil. Thus, the solution of a mixture of castor oil with 5 per cent. of arachis oil becomes turbid at +6° to +5° C.; with 4 per cent. at +3° C., yielding a copious precipitate at 0° C., with 3 per cent. at 0° C.; with 2 per cent. at —2° to —3° C.; and with 1 per cent. it becomes opalescent at —4° to —5° C., and turbid at —9° C. Another method of detecting and estimating arachis and other foreign oils in castor oil is to shake 20 Cc. of the sample with 80 Cc. of petroleum spirit (boiling between 35° and 70° C.) in a stoppered

graduated cylinder, and to allow the liquids to separate. In the case of pure castor oil the increase in the volume of the oily layer, due to solution of the petroleum spirit, will be about 11 or 12 Cc., but in the presence of foreign oils will be greater, and the amount of increase affords a rapid criterion of the purity of the oil. After complete separation, 50 Cc. of the petroleum spirit layer are evaporated and the residue weighed. Twelve samples of pure castor oil yielded from 8.35 to 8.85 per cent. of residue, the average being 8.52 per cent. By deducting the average figure from the amount of residue obtained from a mixture it is possible to estimate the amount of foreign oil present. For example, castor oil containing 30 per cent. of arachis oil yielded 38.61 per cent. of residue; oil containing 4 per cent. yielded 12.73 per cent.; and oil containing 1 per cent. of arachis oil yielded 10.04 per cent. of residue. The residue from the petroleum spirit extract is suitable for the estimation of arachidic acid by Bellier's method.—C. Frabot. (*Ann. Chim. Anal.*, 1917, 22, 217–223.) From *The Analyst.*

Use of Textile Fibers in Microscopic Qualitative Analysis. —Fibers from flax, cotton, viscose silk (cellulose xanthate), lustron silk (cellulose acetate), gelatin silk, wool, mohair, and true silk, were tested with litmus in neutral acid, and alkaline solutions. Silk was found to be the most satisfactory in every respect, the particular kind of silk having little or no effect on the results. The sensitiveness of litmus silk depends on the degree of adsorption of the dye, and on the degree of purification of the raw silk and of the litmus. Silk fibers may also be used as carriers for lacmoid, but this is less sensitive than litmus. The sensitized fibers are examined microscopically for change of color when partially immersed in a drop of the fluid under examination on a slide. Litmus silk fibers can be used for the detection of either acid or alkali, while Congo red viscose silk fibers can only be used for the detection of acidity. In the case of mineral acids a solution as dilute as N/4500 will give a positive reaction with litmus silk; acetic acid fails beyond N/100; the fibers are not quite so sensitive to alkali. Congo red fibers cannot be used to differentiate organic acids from mineral acids. Elaborate directions based on Wartha's procedure (*Ber.*, 1876, 9, 217) are given for the preparation of extremely pure litmus reagent. The most sensitive silk threads for use with litmus were those that had been treated by a preliminary boiling for one hour in a 2 per cent.

"Ivory" soap solution.—E. M. Chamot and H. I. Cole. (*J. Ind. and Eng. Chem.*, 1917, 9, 969–971); through *The Analyst.*

Arsenic in Glucose.—The author made a comparative study of the accuracy of three methods of determining small quantities of arsenic (in solutions free from organic matters), viz., the Marsh-Berzelius, the Gutzeit, and a so-called diaphanometric method in which the arsenic is reduced to the elementary state by hypophosphorous acid in presence of sulphuric acid and estimated from the cloudiness of the liquid in comparison with a series of test solutions of known arsenic-content treated in the same way at the same time. The possible error of all three methods was found to be of the same order, about one tenth of the amount of arsenic determined, but the Gutzeit method is not considered so trustworthy as the others for quantitative purposes. The diaphanometric method is somewhat more expeditious than the Marsh-Berzelius method; it is rendered inexact by the presence of traces of nitrates or nitrites. From solutions containing sugars, traces of arsenic can, after oxidation with bromine, be completely precipitated by entrainment in precipitates of ammonium magnesium phosphate formed in the solutions. Modifying a procedure described by Vuaflart (*Ann. Falsif.*, 1916, 272), the author applied this method to commercial glucoses; the phosphate precipitate was dissolved in nitric acid, all traces of the latter were then eliminated by evaporation with sulphuric acid, and the sulphuric acid solution was used for the determination of arsenic by the diaphanometric method. Experiments described indicate that traces of arsenic in beer-wort are not eliminated to any extent during fermentation or fining of the fermented beer.—J. H. L.—A. Kling. *Ann. Falsif.*, 1917, 10, 438–450; through the *Journal of the Society of Chemical Industry.*

## MEDICAL AND PHARMACEUTICAL NOTES.

Foreign Body in the Eye.—Curran (*Boston Medical & Surgical Journal*, September 27, 1917) states that at the Norton Company during the past year there were treated 1,300 cases of injury to the eye. Of these 90 per cent. were directly attributed to foreign bodies. By the rules of the factory the employees are forbidden to treat each other's eyes, and every man is required to report at the shop hospital for every accident, however trivial. The method of examining the eyes is as follows: First, inspect the lower con-

junctival sac. This can be done by simply pulling down the lower lid and telling the patient to look up, thus exposing the whole conjunctival surface. Then take the upper lid. Here it is necessary to evert the lid in order to expose the whole conjunctival surface. The ease with which this can be done depends upon the experience and consequent dexterity of the surgeon. To do this easily, have the patient look down, grasp the lashes of the upper lid, place an applicator at the base of the tarsal cartilage and evert lid, thus exposing the entire fornix.

The cornea is then carefully searched with the naked eye; with a glass aided by a condensed light. If there be difficulty in the examination this will be remedied by instilling two drops of a 4 per cent. solution of cocaine. Pigment spots in the iris and pigment stains on the cornea may cause some doubt. If a foreign body becomes embedded on the cornea it always leaves a pigment stain, or so-called rust spot. This rust spot disappears in forty-eight hours after removal of the foreign body. A cotton-wound applicator usually removes foreign bodies lying loose in the conjunctival sac. Thereafter 20 drops of a 2 per cent. solution of argyrol are instilled and the patient discharged.

Embedded foreign bodies, whether they are situated on the cornea or sclera, always necessitate the use of an anesthetic, 4 per cent. of cocaine being the best. A corneal spud then enables the operator to remove the body. If the latter be large and deeply embedded it is best to use a Graeffe cataract knife. Thereafter hot compresses are used at home, the patient being given two drops of a 1 per cent. atropine to put the eye at rest. The number of accidents have been reduced to a minimum by the use of goggles, which should always be worn in work involving chopping or grinding or chipping, and where sparks or dust result from the operation. (From the *Therapeutic Gazette.*)

CHAPARRO IN DYSENTERY.—A decoction of *Chaparro amargosa* (*Castela Nicholsoni*) was recommended some years ago for amœbic dysentery (see *Prescriber*, 1914, pp. 225, 284; 1916, p. 118). Chaparro, or bitter bush, is a shrub which grows in Texas and Mexico. The entire plant is used, 6 to 8 ounces of the decoction being given by the mouth half an hour before each meal. Rectal enemata of from 500 to 2,000 Cc. are also given twice daily, these being retained as long as possible. A fluid extract is prepared, the

dose of which is 60 to 180 minims. Its use has been reported in cases where emetine has failed.

In the third annual report of the Medical Research Committee reference is made to this drug by the biochemical department. Preparations of the plant have given promising results, and chemical examination has resulted in the isolation of two crystalline principles, both of which are being clinically tested. (From *The Prescriber.*)

IODINIZED LIQUID PARAFFIN.—Liquid paraffin, as is well known, passes through the intestinal tract unchanged and is not absorbed into the general circulation. This fact suggested to Julius H. Hoelscher (*Therap. Gaz.*, 1917, 33, 689; Oct.) the possibility of using liquid paraffin as a vehicle for drugs whose action it is desired to confine to the intestine, such as internal antiseptics, astringets, vermifuges, etc. His first experiment was with iodine, which was dissolved in the oil in the proportion of one grain to two ounces. The mixture is of a violet color, the iodine apparently being fixed by the oil. Several cases of "intestinal putrefaction" (the term being used for lack of more descriptive words for such cases) were treated, half-ounce doses being given once or twice daily, with satisfactory results. Hoelscher finds that (1) the iodine is not absorbed into the general circulation and cannot be detected in the urine; (2) iodine can be detected in the fæces; (3) there is no indication of iodism or toxic effects; (4) the laxative effect of the oil is not interfered with. He thinks there are great possibilities for a combination of mineral oil and remedies for intestinal parasites (chenopodium, turpentine, thymol, etc.), but this calls for further investigation.

A similar solution of iodine in liquid paraffin is used by R. T. Dobson (*British Med. Jour.*, 1917, 2,717; Dec. 1) as an application in trench foot. (From *The Prescriber.*)

USE OF SOY BEAN AS FOOD.—T. B. Osborne and L. B. Mendel's study shows that the soy bean is relatively poor in calcium and chlorine. The addition of a small amount of calcium carbonate to a food in which the soy bean was the sole source of mineral constituents tended to stimulate growth to some extent, though not so efficiently as the addition of the more complex salt mixture. On diets containing either the soy-bean meal or the commercial soy-

cake meal, together with fats and "protein-free milk" or the authors' "artificial" salt mixture, several broods of vigorous young have been produced, and these young have grown normally on diets the same as those on which their parents were raised. This is a further demonstration of the nutritive efficiency of this legume, in striking contrast with the adverse results obtained with kidney beans and garden peas. The soy bean is the only seed hitherto investigated, with the possible exception of flax and millet, which contains both the water-soluble and the fat-soluble unidentified dietary essentials or vitamins. This fact, taken with the high physiologic value of the protein, lends a unique significance to the use of the soy bean as food. (*Journal of Biological Chemistry,* Baltimore.)

An Effective Treatment for Canine Distemper.—Canine distemper is one of the most prevalent and, with the exception of rabies, the most fatal of all diseases of the dog. It is a disease of the growing dog, but adult animals are not immune to it. In treating it strict attention should be paid to diet, no meat should be given, carbohydrates, dog biscuit, boiled rice, stale bread, vegetables, milk, and meat soup fat strained, may be fed. Tonic drugs such as strychnine (1/500 Gr.), quinine, iron, arsenic, glycerophospates, nuclein, may be used in connection with the hypodermic treatment described below. Acetanilid, aconite, drastic cathartics, and diuretics must be avoided. After numerous experiments on all available medicines, vaccines and antitoxins for the treatment of this condition only two remedies have been found that proved of exceptional value. These are *Echinacea* and *Inula.* The compound of these two was originated five years ago by a New York physician for the treatment of human tuberculosis. It is shown that the two work synergistically to increase the natural resistivity of the blood towards invading organisms much as nuclein does only much more pronouncedly. The phagocytic power of the leukocytes is increased and therefore the opsonic index is raised. The proportion between the red and white corpuscles is normalized by the compound. It acts favorably in leucopenia and hyperleukocytosis; it exerts a certain destructive influence upon the coccus group of microorganisms and effectively eliminates the toxins from the body. For distemper it is to be administered intramuscularly in the thigh every twenty-four hours in doses from 2 to 10 mils according to the size of the dog. If given subcutaneously an abscess is likely to develop. Five

or six injections are necessary alternating on either side of the body. Eighty cases have been treated this way; twelve have died, a mortality of fifteen per cent. No selection of the cases was made, they were taken just as they came into the hospital and many of them were suffering with complications, pneumonia, bronchitis, etc. Dr. S. K. Johnson, of New York, is using the compound in his hospital with like good results. Equine pneumonia, also yield to it. Little says it is the best and most powerful medium by which the opsonic index may be raised, is absolutely non-toxic, and promises to be widely used in many febrile diseases of the respiratory tract, particularly in equine influenza. (George W. Little, D.V.M., Chief Surgeon of the American Society for the Prevention of Cruelty to Animals, New York City. *Am. Jour. Vet. Med.*, 12, 691 (Oct., 1917).)

J. F. C.

Protective Inoculation against Mumps.—The blood of convalescents from mumps was used as a prophylactic. Six to eight mils were injected intramuscularly in 17 children who were exposed to the disease. None of the inoculated children developed mumps, while one third to one half of those not inoculated did so. The blood was taken from children who had just recovered or had been well for ten days. (A. F. Hess, *Proc. Soc. Exper. Biol, Med.*, 12, 144, *C. A.*, 11, 3325.)

J. F. C.

Solution of Soap in Treatment of Wounds.—R. G. Dixon and H. T. Bates have used this dressing in 368 cases. During the same period they treated similar cases with other antiseptics, including eusol, hydrogen peroxid and bismuth iodoform petrolatum paste, with the result that they are using more soap and less of the other antiseptics. A sample of common yellow soap was analyzed and found to contain, per cent.: water, 24.6; fatty acids, 63; combined alkali, 4.6; free alkali, nil; impurities, 7.8. This was considered suitable, and it was found possible to make a 2.5 per cent. solution of it. Small open muscle wounds in which there was no gas gangrene, after being opened up, and metal and cloth removed, were dressed with sterile gauze soaked in this solution. The dressings were left undisturbed for two, three or four days, provided there was no rise in temperature. At the first dressing the most

striking features were less pain, the unusually clean appearance of the wound, absence of pus, and the redness of the whole of the tissues, the muscle in particular. In the same treatment of larger and deeper wounds a 2.5 per cent. solution of the bismuth paste in soft green soap was found to be quite satisfactory. Cases of penetrating wounds of the knee joint were treated by this method, the joints being irrigated and the capsule and external wounds closed. The patients did very well. In cases of gas gangrene the results were usually good. The points claimed for the soap solution dressings are that they clean up a wound quickly, the dressings are much less painful than ordinary dressings, there is a saving of labor, as the dressings need only be changed every three or four days, and the solution is easily procured, easily made and inexpensive. (*Lancet,* London, reprinted from *The Journal of the American Medical Association.*)

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## CORRESPONDENCE.

American Journal of Pharmacy,
145 North 10th St.,
Philadelphia, Pa.

*Gentlemen:* The enclosed is submitted for publication.

At a recent meeting of the board of directors of the Philadelphia Drug Exchange, attention was called, by Mr. H. K. Mulford, to the fact that the British Pharmaceutical Society had worked out (*Lancet,* Nov. 17, 1917, 766) changes in the formulas of the British Pharmacopœia in order to reduce the consumption of sugar and glycerin, that these modified formulas had been officially endorsed and adopted by the Pharmacopœia Committee of the General Medical Council of Great Britain for the duration of the war, and that this body had temporarily withdrawn from the British Pharmacopœia directions for the use of sugar and glycerin in certain preparations.

It is, of course, an open question, whether or not such a revolutionary step as modifying the U. S. P. and N. F. formulas to conserve the national supply of sugar, glycerin and alcohol is necessary *at this time,* as our natural resources are so large, but it is undoubtedly true that, by reason of the enormous demands made upon us, from abroad, sugar, glycerin and alcohol are becoming increasingly

difficult to obtain, commercially, and the time may readily come when it will be impossible to secure such materials commercially and make the official preparations containing them.

The Philadelphia Drug Exchange, therefore, asks the Committee of Revision to consider the expediency or desirability, as a measure of preparedness, so to speak, of framing modified or alternative formulas of U. S. P. IX preparations containing sugar, glycerin and alcohol, and of issuing these in "supplements to the U. S. P. IX" for the duration of the war. The replacements directed should be based not only on conserving the supply, but also in protecting the cost of manufacture. As far as possible the basic ingredients of the formulas should not be replaced by articles of greater cost than those desired to be replaced.

As we understand it, the Committee of Revision of the U. S. P. IX has had the authority given it by the Convention of 1910 to issue "Supplements" to the present revision of the Pharmacopœia (see U. S. P. IX, XXXII), and could issue supplements giving permission to use modified or alternative formulas of official preparations for war emergency use.

The present war is the most destructive in the history of the world, and it behooves us to use every means of preparedness that shall conserve the food and drug supplies of the nation.

Yours very truly,

(Signed) J. W. ENGLAND,
*Secretary, Philadelphia Drug Exchange.*

March 11, 1918.

The following announcement has been sent to the Conference Schools relative to the Fairchild Scholarship.

TO THE SCHOOLS OF THE AMERICAN CONFERENCE OF PHARMACEUTICAL FACULTIES:

*Dear Sirs:* The Fairchild Scholarship this year will be awarded to a graduate pharmacist of one of the Conference schools and the examinations will be held June 25. Kindly make the announcement to your classes so that those who desire can compete for the Scholarship. The examination questions will be sent on order of the dean of your school under seal.

The Fairchild Scholarship Committee will thank you for giving as wide publicity as possible to this announcement.

Respectfully,

E. G. EBERLE,

*Chairman, The Fairchild Scholarship Committee.*

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## NEWS ITEMS AND PERSONAL NOTES.

*F. W. Nitardy, Associated with E. R. Squibb & Sons.*—Mr. F. W. Nitardy, Ph.C., who was recently elected to the office of the first vice-president of the American Pharmaceutical Association, has accepted a position with the well-known pharmaceutical manufacturing firm of E R. Squibb & Sons Co. of Brooklyn and is in charge of a branch of their pharmaceutical productions. Mr. Nitardy was formerly a resident of Denver, Col., and in the employ of the Scholz Drug Co. of that city. He is one of the active members of the A. Ph. A. and is giving efficient service in behalf of pharmacy.

*A Pharmacy Honor Medal.*—Mr. Hugo H. Schaefer, secretary of the New York Branch of the A. Ph. A., proposes a plan for the establishment of a Pharmacy Medal, corresponding in a measure to the "Perkins Medal" and the "Nichols Medal" that are annually awarded respectively by the American Chemical Society and the Society of Chemical Engineering as a distinctive honor for distinguished services rendered by any individual in the respective branches of the science which these national societies represent.

The plan proposed is that the New York Branch shall create a permanent fund to be invested in Liberty Bonds or other gilt-edged securities and the interest therefrom is to be used to defray the expense of the award. The gold medal or bronze medallion, suitably named, is to be awarded annually to the man or woman the results of whose efforts during the preceding year, or whose efforts during a number of years have culminated during the preceding year, would be considered as being the most important and advantageous for American Pharmacy.

The selection of the recipient for the honor is to be made by a standing committee consisting of the past presidents of the American Pharmaceutical Association and in case the number of these living past presidents is less than five, then the senior past vice-

presidents of that Association are to be drawn upon in sufficient number to create a committee of five. No bar is to be placed upon the kind of work accomplished nor is the award to be restricted to any class of pharmacists, i. e., to teachers or so-called professional pharmacists alone.

The medal is to be presented by the senior past president of the New York Branch at a meeting of that Local Branch; the intention being to devote annually one meeting for this special purpose.

*Rev. William Remington an Army Chaplain.*—Rev. William Remington, a son of the late Prof. Jos. P. Remington, who was recently consecrated as suffragan bishop of the Episcopal Church for South Dakota, is now serving in France as chaplain of the Mayo Base Hospital Unit.

*Price Cutting as an Unfair Method of Trade.*—The Federal Trade Commission has cited the United Drug Company to answer a complaint of unfair methods of competition, and has appointed a time for a hearing thereon. It is averred that this company has violated the provisions of the Act of September 25, 1914, by selling at its local stores patent and proprietary articles of merchandise at less than cost and that such action and practices were engaged in for the purpose of suppressing competition in interstate commerce.

*Lieutenant Milson Addresses the Students of the P. C. P.*—Lieutenant Milson of the Canadian Army addressed the senior classes of the Philadelphia College of Pharmacy on Thursday morning, March 14. He very interestingly described the exploits of contingents of the Canadian Army who have rendered such signal service in the present war.

*Adam Hastings Fitzkee, P.D., in the Aviation Corps.*—Lieutenant A. H. Fitzkee, formerly Assistant to the Director of the Pharmaceutical Laboratory of the P. C. P., was a visitor to the College the other day. He has very promptly demonstrated an ability that in the short time since his enlistment has elevated him from the rank of private to a lieutenancy. We expect shortly to hear of his service for the allies on the other side of the ocean and undoubtedly he will give a good account of himself "over there."

*Frank L. McCartney appointed Captain in the Sanitary Corps.*—Mr. Frank L. McCartney, president of the New York Branch of the

American Pharmaceutical Association, and who for the past decade has filled a number of important positions associated with the trade interests, has been appointed Captain, Sanitary Corps, Medical Department of the Army. Captain McCartney has been assigned to service at the Medical Supply Depot at New York City.

*Dr. Frederick B. Power Honored.*—Dr. F. B. Power, who began his career as a research worker while a teacher in the Chemical Laboratory of the Philadelphia College of Pharmacy, his alma mater, and who later achieved an enviable reputation as an investigator mainly in connection with research carried on in the laboratories of Mess Burroughs & Wellcome in England, is serving as a member of the Chemical Committee of the National Research Council which is playing an important role in the present war. He is engaged in the U. S. Department of Agriculture as an expert in pyto-chemistry. Recently he has been further honored by election to the presidency of the Washington Chemical Society and also as Vice-President of the Washington Academy of Sciences.

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## OBITUARY.

Dr. Alfred Birch Huested.—Alfred B. Huested, M.D., Ph.G., until recently professor of botany and materia medica at the Albany College of Pharmacy, died at his home in Delmar, N. Y., on February 23, after an illness dating back to November of last year.

Dr. Huested was born at Clifton Park, N. Y., May 15, 1840. At the age of 12 years he removed to Albany, in which city he continued to reside until 1910, and it was here that his active work in pharmacy was accomplished.

His early education was in the public schools of Albany. At the age of 16, he engaged in the drug business with the firm of Dexter & Nelliger with whom he continued until 1859, when he began the study of medicine.

In 1862 he volunteered and entered the service as hospital steward. Obtaining a short furlough of 20 days during the winter of 1863–1864, he returned to Albany, passed the medical college examinations and that of the State Board. He was commissioned as first lieutenant and assistant surgeon in the 21st New York Cavalry and remained in this service until the summer of 1866. In this year he determined to relinquish the practice of medicine and to

devote his energy to pharmacy and so he established the drug firm of A. B. Huested & Co., in which business he retained an interest until a few years ago.

In 1883, he was elected to fill the chair of materia medica in the Albany College of Pharmacy and retained this position from that time until last November. For the last five years he has, likewise, served as secretary of the Albany College.

In 1884, he was appointed by Governor Cleveland a member of the first Board of Pharmacy of the State of New York. At the organization of the board, he was made president and filled this position for sixteen years and at the time of his decease he was still a member thereof.

Dr. Huested was president of the New York State Pharmaceutical Association from 1880 to 1884. He joined the American Pharmaceutical Association in 1879 and in company with Mrs. Huested was a very faithful attendant at the annual meetings.

THOMAS TYRER.—Mr. Thomas Tyrer, F.C.S., F.I.C., treasurer of the Society of Chemical Industry and engaged in business as the managing director of Thomas Tyrer & Co., Ltd., Stirling Chemical Works of Stratford, died suddenly at his home, Hampstead, London, N. W., on February 21, aged seventy-six. He was born at Wolverhampton and his early education was in his father's school. In 1861, he came to London and entered as a student in the Royal College of Chemistry where in Chemistry he studied under Prof. Hofmann and in biology under Prof. Huxley and physics under Prof. Tyndall.

For more than fifty years he was associated with the manufacture of chemicals and pharmaceuticals. Although not educated as a pharmacist much of his personal work was in the drug and chemical industries closely allied with the drug business. In 1907, the British Pharmaceutical Conference, breaking established precedents, elected Mr. Tyrer as its president; a compliment as deserved as it was appreciated.

He was very closely associated with the work of the Society of Chemical Industry. He served a term as the President of this Society and for many years acted as the Secretary of the London Section of the Society. As a member of the Chemical Trade Section of the London Chamber of Commerce, he took a very active part in the numerous questions of great interest to the development of these industries and the upbuilding of export trade therein.